API Manual of Petroleum Measurement Standards Chapter 14.1

GPA Standard 2166

Collecting and Handling of Natural Gas Samples for Analysis by Gas Chromatography
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Introduction

This standard focuses on gas sampling systems and procedures, recognizing the critical impact of hydrocarbon dew point consideration to the overall accuracy and success of these practices and procedures.

Not all methods are appropriate for all sampling conditions, so it is important to have a thorough knowledge of the phase behavior of the product to be sampled and of the Joule-Thomson effect.

Analyses of gas samples are used for many purposes and are applied to various calculations, some of which have an impact on the accuracy of quantity and quality calculations.

This standard address collection and handling of representative gas samples from streams at or above the hydrocarbon dewpoint, streams that may contain water vapor up to the point of saturation, and streams that may be sour or sweet.

It is not the intent of this standard to recommend particular equipment suppliers or manufacturers.
1 Scope
The purpose of the standard is to provide comprehensive guidelines and procedures for properly extracting, collecting and conditioning a sample from a flowing natural gas stream that represents the composition of the vapor phase portion of the source fluid. This standard considers spot, composite, continuous, on-line and mobile sampling systems and does not include sampling of liquid or multi-phase streams.

2 Normative References
The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any addenda) applies.

GPA 2198: Selection, Preparation, Validation, Care and Storage of Natural Gas and Natural Gas Liquids Reference Standard Blends

3 Terms and Definitions
For the purposes of this document, the following definitions apply.

3.1 Absorption
Occurs when natural gas constituents are dissolved into a liquid or solid that is not considered to be the mixture’s liquid phase.

3.2 Adsorption
Occurs when a thin film of molecules adheres to a liquid or solid surface.

3.3 Chilled mirror test
Used to determine dew points of water and/or hydrocarbon by passing the natural gas over a mirror while gradually reducing the temperature of the mirror until condensation forms. A Bureau of Mines-type of dew point apparatus is commonly used for chilled mirror tests.

3.4 Cricondenbar
The point of maximum pressure on a hydrocarbon dew point curve. This is the highest pressure at which a hydrocarbon mixture can exist separately as gas and liquid.

3.5 Cricondentherm
The point of maximum temperature on a hydrocarbon dew point curve. The cricondentherm is the highest temperature at which hydrocarbon vapor can condense to form liquid.

3.6 Cricondentherm pressure
The pressure at which the cricondentherm occurs on a hydrocarbon dew point curve.

3.7 Continuous sampling systems
Provides for an uninterrupted flow of sample.
3.8
Desorption
Occurs when adsorbed or absorbed molecules are released from a liquid or solid surface.

3.9
Extension tube pigtail
A piece of tubing placed on the end of a sample container used to move the point of pressure drop (point of cooling) away from the sample being acquired.

3.10
Floating piston container
A container which has a sliding (floating) piston that has its forces balanced by a pre-charge pressure.

3.11
Flow-proportional composite sampling
The process of collecting gas over a period of time at a rate that is proportional to the pipeline flow rate.

3.12
Gas sample distortion
Any effect that results in a sample that is not representative of the gas stream or source.

3.13
Gas Sampling Separator
A device in the sampling system used to remove contaminant liquids from the natural gas sample.

3.14
Gas sampling system
The system intended to deliver a representative sample of natural gas from the pipeline to the analytical device.

3.15
Gas quality
Refers to physical characteristics determined by the composition (including non-hydrocarbon components, specific gravity, heating value and dew points) of the natural gas mixture.

3.16
Hydrocarbon Dew Point
The temperature, for a given pressure, at which hydrocarbon vapor condensation begins. For purposes of this publication, the hydrocarbon dew point temperature is considered one of:

- the flowing line temperature (at flowing line pressure),
- the value determined by a Chilled Mirror Test (at flowing line pressure), or
- 30 °F (17 °C) above the calculated hydrocarbon dew point temperature.

3.17
Hydrocarbon dew point curve
A curve connecting all points on a temperature-pressure diagram where a hydrocarbon vapor begins to condense into liquid.
3.18
**Joule-Thomson Effect**
A drop in temperature associated with a drop in pressure. This can result in condensation of heavy hydrocarbons in a natural gas sample. This reduction in temperature is in the order of 7°F (3.9°C) per 100 psi (690 kPa) in pressure drop.

3.19
**Lag time in a sample system**
The time required for a molecule to migrate from the inlet of the sample probe to the inlet of an analyzer.

3.20
**Mobile sampling system**
The system associated with a portable gas chromatograph.

3.21
**Multi-phase flow**
Two or more phases in the stream.

3.22
**Normal condensation**
Caused by an increase in pressure or a decrease in temperature.

3.23
**Normal vaporization**
Caused by a decrease in pressure or an increase in temperature.

3.24
**Phase**
A physical state of a compound (e.g. solid, liquid, or gas). Each phase has a distinct molecular arrangement and can be readily identified (like the two phases of H2O in ice water—solid and liquid).

3.25
**Phase behavior**
Refers to the condensation and vaporization characteristics of a hydrocarbon mixture. It includes considerations such as temperature, pressure and composition.

3.26
**Phase Diagram**
An illustration of the change in dew point and bubble point with changes in fluid pressure and temperature.

3.27
**Pitot probe**
An impact device with an inlet and return port that provides flow to a hot loop by converting velocity into a differential pressure.

3.28
**Re-circulation region eddy**
An area within a piping system where gas is not continually being replaced even though gas is flowing through the system.

3.29
**Representative gas sample**
Compositionally identical, or as near to identical as possible to the sample source stream.
3.30 Residual impurities
Any contaminating substances that are left in a sample container such as air or natural gas components.

3.31 Retrograde condensation
The formation of liquid hydrocarbon in a natural gas mixture as the pressure decreases below the dewpoint pressure.

3.32 Retrograde vaporization
The elimination of hydrocarbon liquids in a two-phase natural gas mixture as the pressure increases above the dewpoint pressure.

3.33 Sample container
Receptacle used for storage and transport of the sample. Typical sample containers are constant volume containers or floating-piston containers.

3.34 Sample path
Part of the sampling system that conveys the sample from the probe to the container or analytical device. It is typically external to the analysis device. This should not be confused with the sample loop that is inside an analytical device such as a gas chromatograph.

3.35 Sample probe
A tubular sample extraction device extending through the meter tube or piping into the stream to be sampled.

3.36 Sample source
Refers to the stream being sampled.

3.37 Single-phase flow
Natural gas flowing at a temperature above the hydrocarbon dew point and free of compressor oil, water, or other liquid or solid contaminants in the flow stream.

3.38 Slip stream, hot loop, speed loop, sample loop
Provides for a continuous flow of sample by circulating a representative fluid of interest through an analytical device (for example) and back to the sampling source.

3.39 Sorption
Absorption and adsorption considered as a single process.

3.40 Strouhal Number
Represents the ratio of inertial forces due to the unsteadiness of the flow or local acceleration to the inertial forces due to changes in velocity from one point to another in the flow field.

3.41 Water dew point
The temperature at a specific pressure where water vapor condensation begins.
4 Hydrocarbon Dew Point

4.1 General

The hydrocarbon dew point is a temperature at a given pressure at which hydrocarbon vapor condensation begins.

In general, when the sample system is kept above the hydrocarbon dew point temperature, all methods, when properly applied, can provide a sample that is representative of the sample source. However, it is difficult (or nearly impossible) to obtain accurate and repeatable results when the temperature of any element of the sample system falls below the hydrocarbon dew point temperature, or when sampling a stream with a temperature near the hydrocarbon dew point.

The hydrocarbon dew point for a particular gas changes with temperature and pressure. Because the hydrocarbon dew point is not constant for all pressures and temperatures, the proper application of the methods in this standard may require the use of a phase diagram. The phase diagram is particularly useful when the pressure or temperature of the gas changes during the sampling process. This tool is discussed in detail in Annex A.

Various equation of state (EOS) computer programs can calculate the hydrocarbon dew point temperature at various pressures (see Bibliographic References 9, 10, and 11). The accuracy of the calculated hydrocarbon dew point temperature depends on the calculation method used and on the accuracy of the gas composition that is entered into the computer program.

4.2 Initial Sampling of a Gas Stream of Unknown Hydrocarbon Dew Point and Composition

In order to properly apply the methods discussed in this standard, it is necessary to start with a reliable value for the hydrocarbon dew point of the stream to be sampled.

For initial sampling of a gas stream of unknown composition, the following techniques are recommended in order of preference:

Measure the hydrocarbon dew point temperature and maintain the sample gas temperature above the measured hydrocarbon dew point temperature.

NOTE- ASTM D1142, Standard Test Method for Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature, recommends that the sample be 30 °F (17 °C) above the dew point.

For the purposes of this standard, the sample should be maintained according to the recommendations in Section 6.12 General Discussion of Heating.

When the hydrocarbon dew point temperature is not measured:

— use a constant pressure spot sampling method while maintaining the sample gas temperature at or above the flowing gas temperature, perform an extended analysis, and calculate the hydrocarbon dew point temperature;

— use a pressure-reducing sampling method, perform an extended analysis, and calculate the hydrocarbon dew point temperature (when using a sampling method involving a pressure reduction, provide sufficient heat, at, or prior to, the point of pressure reduction, to offset the Joule–Thomson effect);

— use historical information, such as analyses or dew point measurements from a similar gas source; or

— take a spot sample at line pressure, heat the sample gas to at least 30 °F (17 °C) above the flowing temperature at the time the sample was obtained, perform an extended analysis, and calculate the hydrocarbon dew point temperature.
NOTE- If the product flowing in the line is completely in vapor phase, the flowing line temperature is above the hydrocarbon dew point temperature for the line pressure. However, if any droplets of liquid have condensed in the flowing product stream, the flowing line temperature is equal to the hydrocarbon dew point temperature for the gas phase at line pressure, the combined liquid and gas product stream is below the hydrocarbon dew point when referenced as a single phase gas product. If the nature of the gas is completely unknown, the safest procedure is to assume that it is at the hydrocarbon dew point temperature. (See Annex A for further discussion)

Once the calculated hydrocarbon dew point temperature is known, maintain the sample gas temperature at least 30 °F (17 °C) above the hydrocarbon dew point temperature as discussed in Section 6.12 General Discussion of Heating.

5 General Considerations for the Design of a Natural Gas Sampling System

5.1 General

The main consideration in the design of a natural gas sampling system is to deliver a representative sample of the gas from the sample source to an analytical device.

Issues that should be addressed when designing a sampling system include: expected gas quality, phase-change characteristics, type of sample/analysis, material to be used for sample delivery, ambient condition extremes, cleanliness, and availability of power, flow rate, and transport time.

Since entrained or free-flowing hydrocarbon liquids are generally collected and accounted for in a condensate recovery system, these liquids are not desired in the vapor phase portion of the accounting system.

Further consideration should be given to the presence of inert compounds such as carbon dioxide (CO₂) or nitrogen (N₂). These compounds will not only impact the heating value and density of the gas, they also may determine the gas chromatograph carrier gas needed.

5.2 The Components of Typical Sampling Systems

Spot and composite sampling methods require the use of a sample container to transport the sample from the field location to the laboratory. Examples are shown in Figures 1a and 1b. Figure 1a is an example of a spot sampling apparatus employing the purging-fill and empty method. Figure 1b is an example of an application using a composite sampler with enclosure.
Figure 1a (left)-Typical Spot Sampling System
Figure 1b (right)-Typical Composite Sampling System within a Heated Enclosure
Figure 2 shows the components in a continuous sampling system or mobile sampling system.

![Figure 2—Typical Continuous (On-line) Sampling System/Mobile Sampling System](image)

NOTE: Insulation and heat tracing details are not shown. See Section 6.12, General Discussion of Heating for gas temperature requirements.

For specific details regarding the design of these systems, see Sections 7.1, 8.1, 9.3.

For details regarding the heating and insulating of sampling systems, see 8.3.4.

6 Flow Characteristics

6.1 General

Piping elements, such as valves and orifices, can create re-circulation regions (eddies) in the flowing sample source stream. The gas composition in these eddies may be measurably different from the gas composition of the bulk flow. The gas downstream of a gas–liquid separator will be near its hydrocarbon dew point, and a reduction in line
temperature will likely cause some condensation to occur. In other cases, a pipeline may be operating with both gas and liquid continually present in the pipe. Obtaining an accurate gas sample under these conditions is difficult and outside the scope of this standard, see Annex C. For a detailed discussion of fluid mechanics, see Annex B.

Users are cautioned not to interpret liquids condensed by the metering or sampling system as free liquids flowing through the pipeline.

6.2 Single-phase Flow

Single-phase flow is natural gas flowing at a temperature above the hydrocarbon dew point and free of compressor oil, water, or other contaminants in the flowing sample source stream. In general, it is preferred that the single-phase gas in the pipeline be in the turbulent flow regime, because the fluid turbulence creates a well-mixed, representative fluid.

6.3 Causes of Gas Sample Distortion

Since natural gas is a mixture of organic and inorganic pure gases, its integrity will be sacrificed if any components of the sample gas are depleted or augmented (e.g. air contamination, desorption of hydrocarbons). This section identifies fundamental mechanisms of sample distortion. An understanding of these mechanisms will reduce the likelihood of poorly designed sampling systems.

It is important to recognize that only sample collection distortion mechanisms are identified in this section. Poor gas analysis techniques can also distort the indicated composition.

6.4 Phase Changes

Condensation and re-vaporization of hydrocarbons within the sampling system can cause significant distortions in a gas sample. These types of sample distortions can occur under both flowing and non-flowing conditions (i.e. in a flowing sample delivery system or in sample containers), or if the sample comes in contact with sampling equipment that is below the hydrocarbon dew point. See Annex A, for a detailed discussion of the thermodynamics of these phase changes.

Accurate sampling from gas streams with temperatures near the hydrocarbon dew point temperature is more difficult than sampling from streams with temperatures above the hydrocarbon dew point temperature. Additionally, depending on the pipeline pressure, retrograde condensation may occur if the pressure is reduced. Some gas mixtures will have a hydrocarbon dew point that is high enough to cause condensation at ambient temperatures commonly experienced during the year. If condensation does occur, and only the gaseous phase of the partially condensed sample is analyzed, the heating value and density of the sample will be incorrect. Furthermore, the remaining sample in the container will no longer be representative of the sample source.

The integrity of gas samples (and calibration standards) with condensed components can be recovered by completely re-vaporizing the condensed liquids, provided that no fluid (gas or liquid) has been released from the sample container prior to re-vaporization. See Annex A.

6.5 Flowing and Sampling Conditions

6.5.1 Overview

A gas sample flowing through a sampling system may experience temperature and pressure changes. Pressure and temperature reductions occur when the gas accelerates through tubing elements within the sampling system. If the gas is near the hydrocarbon dew point, condensation may occur, causing gas sample distortion. It is important to be aware of the fittings and elements within a flowing sampling system, particularly those that cause significant pressure drop. When the nature of the gas is completely unknown, the safest procedure is to assume that it is at the hydrocarbon dew point temperature.
For example, consider natural gas flowing through a restriction such as a partially closed valve or regulator. If the hydrocarbon dew point curve for a gas being sampled is represented by Figure 3, condensation could occur as the pressure of the gas is reduced from 1500 psia to 900 psia (10.3 MPa (absolute)) to 6.2 MPa (absolute)) (path 1–2, Figure 3). It is possible to avoid condensation if the sample gas temperature is maintained above the hydrocarbon dew point. The gas temperature should be high enough to offset the reduction in temperature associated with the pressure reduction (path 1–3, Figure 3). See Annex A for further information.

![Figure 3](image-url)
6.5.2 Sample Conditions

When a non-flowing gas sample, such as a sample contained in a sample container, is subjected to a temperature below the hydrocarbon dew point temperature, condensation (and, therefore, sample distortion) will occur.

A gas sample could condense in the sample container while it is being transported or awaiting analysis in a laboratory. Consider a natural gas with the phase diagram shown in Figure 3 and contained within a sample container. Path 4-5 shows that the sample can condense if the container is exposed to an ambient temperature below the hydrocarbon dew point temperature.

If it is necessary to transfer a small representative sample from a large accumulator, care should be taken to ensure that any condensation that may have occurred has been completely re-vaporized prior to and during transfer and that the sample is well mixed. This is not a recommended practice because it is very difficult to transfer a representative sample from one container to another if the gas is at or near the hydrocarbon dew point.

Gas sample containers and the lines to an analysis device should always be heated prior to analysis. Heating times and temperatures should be sufficient to ensure that any condensed hydrocarbons are completely re-vaporized before an analysis is started. See Section 6.12 General Discussion of Heating and Annex A for further information.

6.5.3 Cold Sampling Equipment

If the sample stream comes in contact with sampling equipment that is at a temperature below the hydrocarbon dew point, condensation, and therefore, sample distortion can occur. Consider the natural gas mixture represented by Figure 3. If the temperature of the sampling equipment is below the hydrocarbon dew point temperature, condensation can occur even when sampling at constant pressure (path 6-7). See Section 6.12 General Discussion of Heating and Annex A for further information.

6.5.4 Surface Effects

There are three important surface effects that have been identified for gas sampling.

a) Clean, solid surfaces are subject to adsorption (sticking) and de-sorption (unsticking) of gas molecules.

b) Some liquids may dissolve gas molecules, or they may yield certain gas molecules if the liquid already contains significant amounts of dissolved gas molecules.

c) Porous surfaces can cause gas sample distortions.

The first two effects tend towards an equilibrium that is dependent on pressure and temperature. If the temperature and/or pressure of the sampling system changes, hydrocarbons could be removed from or released into the sample stream. This will cause an incorrect determination of heating value and density.

Sampling systems shall incorporate clean, inert, and non-porous materials and sufficient temperature control to remain above the hydrocarbon dew point if the surface effect errors are to be avoided.

6.6 Adsorption

Adsorption occurs both chemically and physically. Chemical adsorption is due to a reaction between the gas molecules and the solid surface molecules.

Physical adsorption occurs because free surfaces, such as the inner surfaces of tubing or sample containers, tend to attract gas phase molecules. For a given surface and gas molecule, the extent of physical adsorption depends on the amount of surface area and the pressure and temperature. Changing the pressure and/or temperature could either adsorb or de-sorb gas molecules.
Sample distortion due to chemical and physical adsorption can be minimized by prudent selection of sampling system materials. In general, materials and coatings that are chemically inert and of minimum porosity are the best choices.

6.7 Liquid/Gas Interfaces

Another surface effect that causes gas sample distortion occurs between gas molecules and the surfaces of some liquids. Hydrocarbon liquids can dissolve significant amounts of natural gas components. This is especially important if the sampling system or sample container has been exposed to liquid hydrocarbons.

Proper cleaning procedures will minimize the potential for this problem.

6.8 Plastic Tubing

With the exception of PTFE or PFA or equivalent, most plastic tubing tends to cause gas sample distortion and is not recommended as a gas sample conduit for hydrocarbon analysis.

Be careful not to apply direct heat or high temperature heat tracing to any plastic tubing, including PTFE or PFA

NOTE: PTFE is an appropriate alternative for moisture analyses.

6.9 Residual Impurities

Gas sample distortion can be caused by residual impurities such as air or previous samples and they may be difficult to detect. For example, a gas chromatograph not configured to detect oxygen will show a residual of air as increased nitrogen. This type of impurity might not be detected. If a small amount of the previous gas sample was left in the sampling system or sample container, it might not be detected either.

Strategies for removing residual impurities involve purging, evacuating and repeated fill/empty cycles. Sample distortion due to the presence of residual impurities may be a problem if any of these methods are performed poorly. If there are leaks in any valves or seals these methods may not work, even if the procedures are performed correctly.

6.10 Re-vaporization

Section 6.3 identifies a phase change as one cause of gas sample distortion. This type of sample distortion will have a significant impact on the integrity of the gas sample.

If the gas phase of a partially condensed sample contained in a sample container is analyzed, the heating value and density of the sample will be biased, and the remaining sample will no longer be representative of the sample source.

If a gas chromatograph’s calibration gas standard experiences a phase change and calibration occurs while the calibration gas is still in two phases, all analyses performed from that point forward will be biased. Furthermore, the composition of the calibration gas standard will have changed.

The integrity of the sample (and the calibration standard) can be recovered by heating the containers if no fluid (gas or liquid) is withdrawn prior to re-vaporization. Care should be taken to ensure that all condensed gases are completely re-vaporized when a gas sample is analyzed in a laboratory.

6.11 Cleanliness

Proper cleaning procedures shall be followed in order to remove liquid hydrocarbon residues and other impurities (e.g. water or glycol residue) that may exist.

Sample systems shall be designed so that they may be thoroughly and easily cleaned. A procedure for cleaning sample systems and sample containers is essential for good gas sampling practices. Oil in the transfer lines can
selectively absorb and desorb hydrocarbon components, altering the sample composition. Since new stainless steel tubing may have machine oil from the manufacturing process, sample transfer lines should be cleaned and dried.

Sample containers shall be purged and cleaned prior to each collection of sample, unless they are special passivated containers used to sample streams containing highly reactive components. The most effective cleaning agent is wet steam. Steam cleaning is acceptable only if the steam is clean and does not contain corrosion inhibitors, boiler water treating chemicals, or other substances that may contaminate the sample container.

Solvents, such as acetone and liquid propane, that do not leave a residue after drying are generally acceptable for removing most heavy ends contamination, although they may sometimes present hazards such as flammability and toxicity. Some potassium hydroxide–based liquid detergents also produce acceptable results that compare well to the solvents. Other potassium hydroxide–based solvents can reasonably be expected to produce acceptable results; however, their effectiveness should be tested prior to use.

Supercritical carbon dioxide cleaning produces acceptable results, in spite of the fact that some dirty hydrocarbon spot trails may be left in the container after it is cleaned.

Warning—using supercritical carbon dioxide to clean containers can be hazardous due to the physical properties of the fluid and the high pressures and low temperatures involved and should be used with great caution.

Sample containers shall be dried and purged after wet cleaning procedures (e.g. wet steam). Evacuating the container to 1 mm of mercury absolute (133 Pa (absolute)) (near perfect vacuum) or less will eliminate the residual liquid. Nitrogen, helium, and dry instrument quality air are good examples of gases that may be used to dry or purge containers that are free of deposits and heavy hydrocarbon contamination.

Many laboratories leave a blanket of nitrogen, helium, or other gases in sample containers in order to protect the container from air contamination. Blanket gases and gases used to precharge sample containers shall be carefully selected so that, should leakage occur within the container or should the sample be contaminated by these gases, the chromatograph will not interpret the contamination as a part of the sample being analyzed. For example, a chromatograph using helium as a carrier gas will not detect helium gas left over from the precharge of a single cavity container or helium leaking by the piston in a constant pressure container.

Many sulfur species will be readily absorbed by general purpose sample containers. The resulting analysis will dramatically underestimate sulfur levels. Samples to be analyzed for sulfur content need to be collected in clean and dry specially lined or passivated containers dedicated for that purpose. The entire wetted surface of the sample container and its secondary components (e.g. valves, fittings or relief devices) shall be non-reactive to sulfur or sulfur-containing compounds.

Interaction with water and other components present in the sample gas may degrade the sulfur content regardless of the container material. The sample loop, including any separator in the system, shall be thoroughly purged and be cleared of contaminants and accumulated liquids prior to sample collection.

6.12 General Discussion of Heating

6.12.1 Overview

Condensation may occur in composite, spot, mobile, or on-line sampling systems. If any part of the sampling process causes the sample to cool below the hydrocarbon dew point temperature, scattered and biased analytical results and non-representative samples are likely to result. In order to avoid this problem, the sample gas temperature shall remain above the gas hydrocarbon dew point during sampling. This can be accomplished by heating sample probes and by heat tracing lines, regulators and sample containers or by employing some other means of delivering heat to the fluid in the sampling system.
Due to the uncertainty in measuring or calculating the hydrocarbon dew point, it is recommended that the gas being sampled be maintained at least 30 °F (17 °C) above the expected hydrocarbon dew point throughout the sampling system. This operating temperature margin may be reduced for gas compositions where data (see References 9, 10, and 11) exists to show that differences between calculated dew points and measured hydrocarbon dew points are less than the 30 °F recommendation. If ambient temperatures are above the hydrocarbon dew point, heating may not be required. When the sampling process involves a pressure reduction, provide sufficient heat at or prior to, the point of pressure reduction to offset the Joule–Thomson effect (approximately 7 °F (3.9 °C) per 100 psi (690 kPa) of pressure reduction).

When using a separator or filter to remove unwanted contaminants, the separator or filter shall be maintained at the same temperature and pressure as the gas in the line being sampled. Maintaining the separator at any conditions other than those of the line may cause sample distortion. If the pressure of the separator or filter is at line pressure but the temperature is higher than line temperature, unwanted liquid hydrocarbons may vaporize and artificially enrich (higher BTU value) the sample. If the pressure of the separator or filter is at line pressure but the temperature is lower than line temperature, portions of the vapor phase may condense, be removed by the separator or filter, and artificially deplete (lower BTU value) the sample.

Generally, additional heat is not required when using the evacuated container, helium "pop", water displacement, glycol displacement or piston container methods. When additional heat is required, the system shall be heated from the sample source outlet valve through the inlet valve of the sample collection container.

When using the purge, fill and empty or continuous purge methods and additional heat is required, the system shall be heated from the sample source through the outlet valve of the sample collection container.

When using the reduced pressure, portable or on-line gas chromatograph methods and additional heat is required, special attention should be directed to the pressure reducing devices to ensure the gas is maintained above the expected hydrocarbon dew point (see section 12.1 NOTE) throughout the sampling process. The system shall be heated from the sample source through the pressure-reducing device to prevent condensation due to the Joule-Thomson Effect and downstream of the pressure-reducing device if ambient conditions require. If external filters are used after pressure reduction or separation, the filters should be operated above the hydrocarbon dew point.

### 6.12.2 Heat Tracing

Heat tracing methods most commonly used are steam, hot water and electrical.

Electrical heat tracing is normally used in remote locations or in locations where it is difficult to use steam or hot water. Electrical heat tape should be self-limiting or controlled with temperature-limiting devices.

Electrical heat tracing and all electrical equipment should meet the electrical codes for the intended service area. These requirements ensure that a heating element does not overheat if a failure in the electrical components occurs. Overheated electrical components could cause injury or an explosion in a natural gas application. Under no circumstance should any component of the sampling system, such as the sampling probe, regulator, tubing, valves, filter, regulator or sample container, be heated above manufacturer's recommended maximum temperatures.

Sampling systems on residue gas lines on the outlet of gas plants should be heat traced to ensure that liquids do not condense in the sample system during plant upsets. Once these liquids accumulate in a sampling system, it may take days after the process is operating normally before representative samples are collected from the system. Purging the entire system may be required.

### 6.12.3 Catalytic Heater

A catalytic heater produces heat through an exothermal reaction between a combustible gas, such as natural gas, oxygen and a catalyst. The heat given off by the reaction can be adjusted by varying the rate of gas flow to the catalyst. The heat released by the reaction is well below the ignition temperature of the natural gas.
Electrical heat tracing and all electrical equipment should meet the electrical codes for the intended service area. These requirements ensure that a heating element does not overheat if a failure in the electrical components occurs. Overheated electrical components could cause injury or an explosion in a natural gas application.

6.12.4 Insulation

Insulation is used to protect the flowing stream from cold ambient conditions or covering heat tracing around the external portions of the probe assembly, regulator and sample line. It will help ensure that the stream being sampled remains above the hydrocarbon dew point during the sampling process.

7 Sample Probes

7.1 General Design Considerations

Sample probes are designed for the purpose of directing a representative portion of the natural gas sample source into the sampling system. There exists, along the pipe wall, a laminar flow area which may not represent the product that is flowing through the pipe. At normal flow velocities, this laminar flow section is very thin. The Sample Probe should extend into the pipeline to help ensure that the sample is withdrawn from the turbulent flow region. The probe also extends into the pipeline to ensure a representative sample that is free of unwanted contaminants such as oil, water, amine, glycol, etc. that tend to flow along the inside wall of the pipe. Sample probes may be designed as fixed or as insertable and retractable units. A well-designed sampling system requires the use of a properly installed sample probe. There are several designs of sample probes available. The design should also consider the possibility of resonant vibration being induced in the probe by high flowing velocities in the pipeline. Gas lines with streams free of entrained liquids and at flowing conditions well above their dew point temperatures may be sampled with any probe design. Lines that are operating at or near the gas stream’s dew point may require special probes designed to overcome the problems of condensation in the gas. In selecting a sample probe, the first step is to determine sampling conditions and then to determine the proper sample probe for the application.

7.2 Application

Sample probes and other components of sampling systems should be designed to extract and deliver a representative sample of the sample source.

7.3 Types

7.3.1 Straight Tube Probes

The most basic sample probe design is the straight tube probe shown in Figure 4. The probe may be attached to a fixed coupling assembly or to one that allows the probe to be removed completely. All fittings installed in the sampling system should be non-restrictive and should be rated for the intended line pressure and temperature. The materials should be suitable for use with the product, its contaminants and ambient conditions. Modifications to this basic design are numerous. Filters and screens added to the collecting end of the probe may reduce the possibility of small liquid particles entering the probe. This practice should not alter the composition of the sampled gas. Inappropriate probes or filters could alter gas streams that are very close to the hydrocarbon dew point. The collection end of the probe may be straight or angle-cut. Straight-cut probes are preferred over beveled probes. If the probe is diagonally cut, it should be oriented such that the longer part of the diagonal is facing upstream. With this configuration, small droplets entrained in the product stream will generally have enough inertia to pass by the probe without being withdrawn into the sample system.

Flow disturbances may cause contaminants to form aerosols that can be ingested by the sample probe. These aerosols should either be excluded by the probe or removed in the sample system by separation or filtration.
Pressure regulators can be part of the sample probe (Figure 5). This allows heat from the flowing product stream to assist in heating the sample back up to sample point temperature. The flow rate through the probe regulator affects the recovery of product temperature. Regulating probes are commonly used with continuous sampling systems designed to deliver gas to the sampling system at reduced pressure. They may also be used to reduce pressure to keep the stream below the hydrocarbon dew point pressure in some cases. The probe’s most basic form is a straight tube with an integral regulating mechanism.

If source conditions are such that retrograde condensation is possible, then condensation of the sample gas could occur in the probe after the pressure is reduced, and a non-representative sample will result. Fins may be used on the probe to improve heat transfer with the sample gas to prevent condensation. Care should be taken to minimize the effect of thermally coupling the probe to the pipe carrying the sample source stream.

Figure 4—Straight Tube Sample Probe

7.3.2 Regulating Probes
7.3.3 Membrane Filter Probe

The membrane filter probe may be directly mounted in the product stream and may or may not have pressure regulation as an integral part of the probe.

In cases where an integral regulator is not sufficient, a heated regulator may be mounted directly on the outlet of the probe, heating the sample to a temperature greater than 30°F (17°C) above the hydrocarbon dew point, which presents a sample to the sample container inlet valve that is comfortably in the vapor phase and representative of the vapor phase of the flowing gas stream.

7.3.4 Pitot Probes/Dual Probes

A Pitot probe can be used provided the gas velocity in the pipeline is sufficient to drive the flow throughout the entire loop, or two straight tube probes can be installed across a differential pressure point, assuring flow throughout the system.

NOTE- Adequate data is not available to make recommendations regarding the performance of Pitot probes.

7.4 Probe Installation

7.4.1 Sample Probe Length

Since free-flowing liquids tend to flow along the bottom of the pipe or flow along the walls of the pipe, it is necessary to have a sample probe extending into the pipe. To prevent failure of the sample probe in the flowing gas stream, sample probe length should be considered with care.

A sample probe should be used for all sampling techniques. The collection end of the probe should be placed within the approximate center one-third of the pipe cross section. However, under no circumstances should the sample probe be longer than 10". While it is necessary to avoid the area most likely to contain migrating liquids, i.e. the pipe wall, it may also be necessary to limit the probe length to ensure that it cannot fail due to the effects of resonant vibration.
Resonant vibration can occur when the vortex shedding frequency resulting from a probe inserted into a flowing fluid is equal to the probe's natural resonant frequency. Table 1 provides maximum recommended probe lengths for typical diameters based on a maximum natural gas velocity of 100 ft/s (30.48 m/s)

Caution: Harmonics may cause embrittlement of the metal. Poorly designed sample probes may bend or break off in the flowing gas stream.

Table 1—Maximum Recommended Probe Lengths

<table>
<thead>
<tr>
<th>Probe Outer Diameter inches (cm)</th>
<th>Recommended Max Probe Length inches (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.25 (0.64)</td>
<td>2.00 (5.08)</td>
</tr>
<tr>
<td>0.375 (0.95)</td>
<td>3.25 (8.26)</td>
</tr>
<tr>
<td>0.50 (1.27)</td>
<td>4.25 (10.80)</td>
</tr>
<tr>
<td>0.75 (1.91)</td>
<td>6.50 (16.51)</td>
</tr>
</tbody>
</table>

Calculations were based on a maximum recommended probe length Strouhal Number of 0.40, a 0.035 in. (0.089 cm) wall thickness, and 316 stainless steel probe construction. For conditions other than these use the formula below to calculate the maximum recommended probe length.

The probe length is defined as the distance between the probe tip and its point of attachment. The probe depth is the distance between the probe tip and the inner pipe wall (Figure 6).

**7.4.2 Probe Length Calculation SI Units**

The maximum insertion probe length (from the end of the probe to the point of attachment) can be calculated (from EEMUA Publication No. 138:1988) using the following formula:

\[
L = \frac{[(Fm \times 4.38 \times OD \times 10)/(S \times V)] \times ((E/r) \times (OD^2 + ID^2))^{1/2}}{(E(r) / (r^2))^{1/2}}
\]

Where:

- **L** = permissible probe length (mm)
- **Fm** = Virtual mass factor: a constant to take account of the extra mass of the container due to the fluid surrounding it and vibrating with it. For a gas, Fm = 1.0 and for water and other liquids Fm = 0.9
- **OD** = Outside Diameter of the probe (mm)
- **ID** = Inside Diameter of the probe (mm)
- **S** = Strouhal number- dependent on the Reynolds number and the shape of the container but can be taken as 0.4 for worst case or 0.2 as suggested by API MPMS Chapter 8.
- **V** = Velocity of fluid (m/s)
- **E** = Modulus of Elasticity of probe material (kg/cm²)
- **r** = Density of probe material (kg/m³)

**7.4.3 Probe Length Calculation US Customary Units**

The maximum insertion probe length (from the end of the probe to the point of attachment) can be calculated (based on EEMUA Publication No. 138:1988) using the following formula:
\[ L = \left[ \left( \frac{F_m \times 1.194 \times OD}{S \times V} \right) \times \left[ \frac{E/r \times (OD^2 + ID^2)}{2} \right] \right]^{1/2} \]

Where:

- **L** = Permissible Probe Length (inches)
- **Fm** = Virtual mass factor - a constant to take account of the extra mass of the container due to the fluid surrounding it and vibrating with it. For a gas, Fm = 1.0 and for water & other liquids Fm = 0.9
- **OD** = Outside Diameter of the Probe (inches)
- **ID** = Inside Diameter of the Probe (inches)
- **S** = Strouhal number - Use 0.4 for worst case
- **V** = Velocity of fluid (ft/s)
- **E** = Modulus of Elasticity of probe material (psi)
- **r** = Density of probe material (g/cm³)

![Figure 6—Probe Dimensions Used to Determine Maximum Recommended Probe Length](image)

7.4.4 Sample Tap Location

Refer to the appropriate AGA or API measurement standards pertaining to meter tube design for probe placement restrictions.

Probes should be mounted vertically at the top of a straight run of horizontal pipe. Care should be used in selecting the location of sampling points. Flow-disturbing elements such as control valves, headers, elbows, tees and orifice plates can cause pressure and temperature disturbances in the flowing gas stream immediately downstream of the disturbance. It is important that the sample point be separated from flow disturbing elements far enough to avoid the areas of flow disturbance. For streams that are at or near their hydrocarbon dew point (either continuously or only during process upsets), the probe should be at least five diameters (of the maximum diameter of the disturbing element) downstream of that disturbance. For major disturbances that change the flow profile of the flowing stream
(such as orifice plates, elbows, tees, reduced-port valves, flow conditioners, filters, strainers, etc.), the diameter of the disturbance shall be considered to be the inside pipe diameter at the disturbance. This requirement is intended to avoid the effects of liquid droplets or liquids formation due to the impact of the flowing stream on the disturbing element. Similar precautions should be taken for streams with liquid contaminants such as glycols, amines, compressor oils, corrosion inhibitors, etc.

Example 1, if a temperature probe has a diameter of 1", the sample probe should be at least 5 temperature probe diameters or 5" downstream of the temperature probe.

Example 2, a sample probe in a 10" ID line would need to be at least 50" downstream from a major disturbance.

Probes should not be installed in any “dead-end” section of pipe, where gas is not continually flowing or where there may be recirculation regions or “eddies.”

8 Sample Loops and Transfer Lines

8.1 General Design Considerations

The sample loop (slip stream) is the part of the sampling system that delivers the gas from the sample probe to the inlet of the sampler or analysis device and then to a lower pressure point. Sample loops should be designed to deliver a representative sample of the sample source gas that is flowing in the pipeline. The velocity of the gas flowing in the sample line and the volume of the sample system determine how often a new representative sample can be obtained. It is preferred that the sample line be sloped upward from the sample probe to the sample extraction point. This will minimize the accumulation of liquid in the loop.

In order to obtain a representative sample, the loop should be designed to ensure that the volume of gas in the sample loop is replaced between samples. This requires the sample loop flow rate to be relatively high and the volume of the loop to be small. Excessively high flow rates may cause liquid particles present in the pipeline to be drawn into the sample probe. Sample loops that purge to the atmosphere can cause unacceptable amounts of gas waste and violate environmental regulations. In addition, a large pressure loss in a sample loop may cause cooling and condensation which will affect the accuracy of the sample.

No component in the sample system should be heated or pressurized above manufacturer's recommendations.

8.2 Pressure Drop in a Sample Loop

Proper operation of a sample loop requires a pressure differential from the collection end to the discharge end. This pressure differential may be produced with an orifice plate, regulator, or pump. Attaching the ends of a sample loop across a flow restriction will provide a pressure differential to the sample loop that is proportional to the flow rate squared. This arrangement will provide a flow in the sample loop that is proportional to the flow through the orifice. Custody transfer meters cannot be used for this application because the flow through the sample loop will bypass the meter and result in a biased flow rate measurement.

8.3 Tubing

8.3.1 Material

A sample transfer line should be non-reactive to the sample. It should be rated for the maximum expected pressure and should retain its strength at the maximum or minimum expected temperature. Stainless steel (304 or 316) and PTFE or PFA have been proven to be non-reactive to natural gas components. Stainless steel is the recommended material under most conditions and is preferred for pressures over 1000 psig or for gases containing hydrogen sulfide (consider silica or other non-reactive material-lined stainless steel tubing for hydrogen sulfide applications, as stainless steel is known to adsorb H₂S).

PTFE or PFA or an equivalent can be used if the product sampled adversely effects or is affected by stainless steel tubing. It can also be used effectively in certain applications where excessive heat is not required. It is particularly useful when checking for the presence of free liquids.
8.3.2 Diameter

Tubing diameter should be as small as possible while ensuring adequate flow through the system. Diameters of \( \frac{1}{16} \), \( \frac{1}{8} \), and \( \frac{1}{4} \) in. (1.59 mm, 3.75 mm, and 6.35 mm, respectively) have relatively small volumes per unit length that allow free liquids to be more easily swept out of the loop. If mechanical security is a concern, the tubing can be encased in larger tubing or pipe.

For spot sampling, the transfer line should have a minimum diameter of \( \frac{1}{4} \) in (6.35 mm). To ensure that refrigeration associated with the Joule-Thomson Effect occurs some distance downstream of the sample container, the smallest diameter component in the spot sample system should be the orifice in the drilled plug at the end of the extension tube ("pigtail") attached to the outlet of the sample container. The drilled plug can be replaced with a flow control valve provided that the internal diameter of the flow control valve is smaller than any other component in the sampling system.

For the portable and on-line gas chromatograph method, the sample transfer line would generally have a diameter of \( \frac{1}{16} \) inch (1.59 mm) to \( \frac{1}{4} \) inch (6.35 mm). For the portable or on-line gas chromatograph method, sample transport lag time calculations are helpful in determining appropriate sample line diameter.

8.3.3 Length

The sample transfer line should be as short as possible. Inches (cm) are preferred to feet (meters). Short lines reduce the heat transfer with ambient surroundings. If ambient temperatures are lower than the temperature of the system being sampled, cooling of the sample tubing can cause condensation of the heavier (higher boiling point temperature) components. These condensed components can then drop out or adhere to the wall, causing the sample to be lighter (fewer heavy components, lower heating value). Or the accumulated droplets can be swept into the sample container, causing the sample to be heavier (higher heating value) than the product to be sampled.

For the portable or on-line gas chromatograph method, sample transport lag time calculations are helpful in determining appropriate sample bypass purge rate. The below formula will allow the calculation of the actual sample transport time.

\[
t = \frac{VL}{F_s} \left( \frac{P + 15}{15} \right) \times \left( \frac{530}{T + 460} \right) \times \left( \frac{1}{Z_p} \right)
\]

Where:
- \( t \) = Purge Time
- \( L \) = Line Length, ft
- \( V \) = Tubing Volume, ccf
- \( F_s \) = Standard Flow, ccf/min
- \( P \) = Actual Pressure, PSIG
- \( T \) = Actual Temperature, °F
- \( Z_p \) = Compressibility at P Pressure

* This formula was taken from Appendix F; pp 579 and 580; "Process Analyzer Sample Conditioning System Technology" by Robert E. Sherman; Wiley Interscience, and modified for Gas Compressibility by ABB Totalflow Project Engineering.

8.3.4 Insulation and Heating

Sample transfer lines should be maintained above the hydrocarbon dew point. For very short connections between the sample point and the sample container, the sample transfer line may be insulated without supplemental heating, but care should be taken to ensure that this short transfer line is at the same temperature as the sample source prior to transferring sample to the sample container.
8.3.5 Cleaning

All sample loop tubing should be steam cleaned and dried before installation. Any oil left in the tubing from the manufacturing process should be removed. This oil can retain or release some of the heavier hydrocarbon components. The amount of components retained or released depends on their concentration in the sample gas, the sample loop temperature, the flowing pressure and the amount of the tubing coated with oil.

The sample loop should be cleaned if free liquids have or are suspected to have contaminated the system.

8.4 Pressure Regulators

Pressure regulators are often required at the point where the gas sample is withdrawn from the sample loop, to reduce the gas pressure from pipeline pressure to a usable pressure for the sample container or analysis device. Specialized pressure regulators that are inserted into the pipeline to take advantage of the flowing gas temperature are available (see 7.2.2). Insertion type regulators that incorporate integral filtration to remove free liquids are also available. Pressure regulators should have a pressure rating that exceeds the maximum expected line pressure of the gas sampling system. Regulators should be constructed of materials that are not reactive with the gas being sampled.

Care should be taken when using any type of regulator, to ensure that the gas does not condense. Retrograde condensation may occur even if the gas is maintained at pipeline temperature. If the gas changes phase and condensation does occur, a representative sample will not be obtained.

One method of reducing the possibility of heavy end condensation in a pressure regulator is to use a heated regulator. This type of regulator should be designed to supply enough heat during the pressure reduction to avoid condensation. Another method of reducing the possibility of condensation is heat tracing. In general, the amount of heat energy required to offset the effect of the pressure drop will depend on the gas composition, pressure, temperature, pressure drop and hydrocarbon dew point. The gas temperature should be maintained at a temperature high enough to offset the temperature reduction associated with the pressure reduction.

8.5 Pumps

Pumps may be used to provide sufficient gas flow through a sample loop. The pump should be installed to provide steady flow without significant pulsation or flow interruptions. The pump produces the best results when it is installed downstream of the sample container or analysis device. The pump and the sample loop line size should be properly matched to assure that pump damage will not occur. The preferred pump for the application is a centrifugal pump.

8.6 Filters

Research has shown that misuse of separators and filters can cause sample distortion.

It is important for all sample system components to be clean and free from contaminants. This is equally true for filters. Refer to Annex I, Cleaning for cleaning procedures and for procedures to evaluate the effectiveness of cleaning procedures.

Particulate filters are used to remove solid, abrasive particles from the sample source stream. This is primarily intended to protect the analysis equipment. Typical filter sizes are 2 to 7 μm (0.08 to 0.28 μl) and generally do not affect analytical results more than ±0.25 %.

Be sure to change filters regularly and ensure condensation does not occur within them which can alter the analysis.

Multiple streams with different heating values flowing through the same filter may cause sample distortion and should be avoided.
It is imperative that the temperature and pressure of filters that are used as a separator (installed upstream of any pressure-reducing regulator) match the flowing natural gas temperature and pressure throughout the sampling process.

Filters that are installed in the sample system after a pressure-reducing regulator should be operated above the hydrocarbon dew point. The Joule-Thomson effect from the pressure drop across a filter should also be considered when determining the heating requirements.

External filters may be used to eliminate contaminants from the sample stream. If the filter is operated at line pressure, it should be operated at line temperature. If it is above line temperature, it may vaporize liquid contaminants to artificially enrich the sample. If it is below line temperature, it may condense components to artificially reduce heavy components in the sample.

Some filters or screens may encourage the creation of liquids not otherwise present in the stream when operating at conditions near the sample dew point. If external filters are used after pressure reduction or after a separator or membrane filter, the filters should be operated above the hydrocarbon dew point.

For some sampling methods, the pressure is reduced at the sample point and the temperature of the sample system after the regulator is maintained at least 30°F (10°C) above the calculated hydrocarbon dew point throughout the sampling process.

On-line and portable chromatographs frequently have filters installed at the sample inlet. Filters in such systems should be maintained at least 30°F (17°C) above the calculated hydrocarbon dew point throughout the sampling process.

An acceptable method for removal of unwanted contaminants is a membrane filter inserted directly in the line (See Figure 5). This type of filter is, by virtue of its location in the pipe, at the same temperature and pressure as the sample source. This eliminates condensation or vaporization of heavy components due to changes in temperature or pressure. In special circumstances defined by some governmental regulations, membrane filters integrated into the sample probe might not be permitted. In such cases, any filtration would need to be performed external from the probe.

It is imperative that the temperature and pressure of filters that are used as a separator (installed upstream of any pressure-reducing regulator) match the flowing natural gas sample source temperature and pressure throughout the sampling process.

8.7 Separators

It should be noted that in streams outside the scope of this Standard, gas separators (aka GPA Separator) may be used to prevent free liquids from entering the section of the sampling system downstream of the separator.

Research has shown that misuse of sample line separators can cause sample distortion. Refer to Annex J.

9 Sample Containers

9.1 General Design Considerations

A sample container stores a gas sample in a protected and secure state until the gas composition can be determined. All mechanical components such as container material, valves, seals, lubricants and other components of the container shall not alter the gas composition. In addition, container used for sampling shall be clean and free of leaks. Cleaning and leak checks of container and components shall be carefully performed prior to each use to ensure that no contamination of the gas sample will occur.
Sample containers shall be labeled with an identification number and maximum working pressure. If required, the date of the last physical inspection should either be included on a container label or be readily accessible. If the container is to be transported, it should meet U.S. DOT (CFR 49) specifications.

During sampling, the container temperature shall be kept above the hydrocarbon dew point. If the sample container is exposed to temperatures below the hydrocarbon dew point after the sample has been collected, the sample can be recovered by heating it to a minimum of 30 °F (17 °C) above the flowing temperature at the time the sample was obtained and maintaining the sample container at that temperature for at least two hours prior to analysis. If a sample is collected at a pressure other than line pressure at the time of collection, the sample shall be heated at least 30 °F (17 °C) above the calculated hydrocarbon dew point of the sample at conditions in the sample container at the time of collection. (See Section 6.12 General Discussion of Heating for additional guidance regarding the operating temperature margin of 30 °F (17 °C) above the calculated hydrocarbon dew point.)

Sample containers can be heated using a water bath, heating blankets, heat tape, or a heated chamber as long as the temperature of the heating medium is controlled. Heat lamps and similar devices are not recommended since it is difficult to control the temperature of the container. Any method used for heating the sample containers shall meet the requirements of all applicable codes and regulations.

Whenever a sample container assembly is heated above 125 °F, special care shall be taken to consider the effect of the heat on seals and other materials. Use the sample contents/transfer label to verify that the contents of the container will not overpressure the sample container when heated. Exercise care to avoid heating containers filled with liquids, as the container may be over pressurized.

9.2 Types of Sample Containers

9.2.1 Single- and Double-valve Standard Containers

These containers are also known as constant volume containers, single cavity containers, and "spun" containers. Examples are shown in Figure(s) 7.

Use metal sample containers of a type that ensures maximum safety and designed to safely meet the anticipated operating extremes for their intended service. Containers and components should be non-reactive and corrosion resistant to the product being sampled. Stainless steel containers are recommended to minimize problems of surface absorption and/or adsorption of heavy components (hexanes and heavier components) and to minimize the reaction of contaminants with the container. If the container is to be transported, it should meet DOT specifications and be labeled according to DOT hazardous materials regulations and applicable state regulations. See Section 15.

Depending on the sampling procedure selected the container may be of the two-valve (Figure 7a) or one-valve type (Figure 7b). Sample containers and valves should have a working pressure equal to or exceeding the maximum pressure anticipated in sampling, storage or transportation of the sample container. Soft-seated valves are preferable to those having metal-to-metal seats. All valves and safety devices should meet the appropriate material and pressure requirements for safe design. The pressure relief valves may be of spring or rupture-disc type. These allow a partial or complete loss of contents due to thermal expansion or over pressurization. The safety device should indicate if it has operated. Should relieving occur, the sample is likely to be compromised and should be discarded. For some containers, a pressure relief device may not be required, refer to applicable transportation regulations. The size of the container depends both upon the amount of sample required for the laboratory tests that are to be made and the volume required to obtain a representative sample from composite sampling systems based on time or flow.
Figure 7a—Typical Double-Valve Sample Container
9.2.2 Floating Piston Containers

These containers are also known as constant pressure containers. An example is shown in Figure 7c.

A floating piston container is constructed of metal tubing, honed and polished on the inside surface. The container should be closed with removable end caps to provide access to remove and service the moving piston. The end caps are drilled and tapped for valves, gauges, and relief valves. The complete container assembly should be designed to withstand the maximum pressure and temperature anticipated during sampling, transportation and analysis and to be non-reactive to (a) materials being sampled, (b) the pressurizing fluid, (c) the cleaning solvents, and (d) the expected corrosives. The volume of the container will depend on the amount of sample needed for the laboratory analysis. If the container is to be transported, it should meet DOT specifications and be labeled according to the federal hazardous materials regulations and applicable state regulations. See Section 15.

The container itself contains a moving piston equipped with O-rings, PTFE rings, or other devices to create a leak-free seal between the sample and the inert, back-pressure gas and still allow the piston to move freely within the container. The use of guide rings is recommended to ensure smooth piston travel. The piston and sealing device should be nonreactive to (a) the sample, (b) the back-pressure gas, (c) the cleaning solvents, and (d) expected corrosive components in the gas.

All valves and safety devices should meet the appropriate material and pressure requirements for safe design. The pressure relief valves may be of spring- or rupture-disc type. These allow a partial or complete loss of contents due to thermal expansion or over pressurization. Should relieving occur, the sample is likely to be compromised and should be discarded.

Some piston-type containers are fabricated from nonmagnetic materials such as 300 Series stainless steel. The piston is likewise fabricated of stainless steel, but has magnets attached to the precharge side of the piston. As the piston moves the length of the container, the magnetic field generated by the magnets flips a series of bicolored flags. This system (or systems of similar configurations) indicates the piston position and volume of sample in the container.
Some piston-type containers are fabricated with a rod attached to the piston that extends through the end cap on the inert gas back-pressure chamber with appropriate sealing devices to prevent the inert gas back-pressure from leaking. The travel rod provides an indication of the piston position and the volume of the container filled with the sample. Again, some modifications of this style may exist.

Some types of constant pressure containers are equipped with electronic tracking devices to provide for local and/or remote indication of the piston’s position relative to full/empty.

Figure 7c—Typical Floating Piston Container
Other types of floating piston containers are available which have no visual method of determining the sample volume directly. For these containers, a magnet or some other type of locating device is necessary to follow the movement of the piston.

9.3 Pressure Regulators and Regulating Probes

The gas temperature should be maintained at a temperature high enough to offset the reduction in temperature associated with pressure regulation (approximately 7 °F [3.9 °C] per 100 psi [690 kPa] of pressure reduction). If necessary, heat the regulator or regulating probe and the associated tubing (where exposed to ambient conditions) to maintain the gas temperature at least 30 °F (17 °C) above the calculated hydrocarbon dew point temperature.

9.4 Composite Sampling Systems

It is recommended that these systems, including the sample container, be maintained above the hydrocarbon dew point to ensure the composite sample is representative of the flowing gas stream. Tests conducted under actual field operating conditions have shown that composite sampling systems do not consistently provide representative samples when exposed to ambient temperatures below the sample gas hydrocarbon dew point.

NOTE – Adequate data is not available to determine the impact of sample containers being below the hydrocarbon dew point and all other components of the composite sampling system above the hydrocarbon dew point.

9.5 Calibration Standards

Refer to GPA 2198 for proper handling of calibration standards.

10 Materials for Sweet and Sour Gas Service

10.1 General Considerations

The types of materials used in a sample system will depend on the gas being sampled. Generally, it is recommended that 304 or 316 stainless steels be used for all wetted surfaces. Valve seats, O-rings and piston seals should be made of elastomers appropriate for the intended service. Sampling of H2S, CO2, wet- and high-temperature gases, present additional material problems. These types of gases will sometimes require special materials and coatings in the sampling system.

It is recommended that sample containers used in sour and/or corrosive gas service should be specially lined or coated (e.g. epoxy). Occasionally, sample containers may be glass or ceramic-lined; however, such containers may be absorptive or adsorptive under certain conditions. Other coating materials and/or passivation may be acceptable. Very reactive components, such as hydrogen sulfide (H2S), should be analyzed onsite when practical since even coated containers may not eliminate all absorption or reaction of the contaminants.

The use of soft metals such as brass, copper, and aluminum (except hard anodized) should be avoided in a sample system because of excessive corrosion rates and other metallurgical and sampling problems. Corrosion rates and the possibility of sulfide stress corrosion cracking for each sampling system should be considered and the service life of the equipment reduced to account for corrosion. NACE standards or other appropriate standards for materials should be applied to the containers and sampling systems.

10.2 Carbon Steel

Carbon steel and other relatively porous materials may retain heavier components and contaminants such as CO2, N2, and H2S in the natural gas sample source stream and should not be used in a sampling system. Reaction of carbon steel with the components frequently found in natural gas will cause errors in the gas analysis. Sample valves and containers made from this material display dangerously high corrosion rates. These problems are particularly
acute in wet sour gas sampling. Corrosion rates in a carbon steel sample system may be sufficient to cause particulate contamination of the sampling system valves, filters and analysis equipment.

10.3 Dissimilar Materials

Using dissimilar materials in a sample system may cause increased rates of corrosion and may result in sampling errors. When taking several samples of a gas for comparative analysis, the same container material should be used. Using the same material reduces the likelihood that the gas will react differently with each sample container but does not guarantee that the sample will not be distorted.

11 System Considerations

11.1 General

The sample delivery system may require heating and insulation to avoid sample condensation. The system should also be designed to facilitate cleaning and the sample lines should be kept short as possible. The flow rate through the sample system should be designed to achieve the appropriate lag time in the system. For example, in real-time process control, the lag time should be very short relative to the requirements for a monthly average analysis.

11.2 Timers

Timers are used on time-proportional sampling systems to actuate the sample system and collect a sample at the desired intervals. See Section 13.

11.3 Flow Computers

Flow computers or flow indicators may be used on a sample system to provide an indication of the flow rate to a flow-proportional sample system. Flow computers and flow indicators should meet all appropriate electrical standards for the areas of their intended use.

11.4 Power Supplies

Systems requiring a power supply should meet appropriate electrical standards and should have a backup power source available in case the primary power source fails.

12 Spot Sampling Methods

12.1 General

The procedures recommended for spot sampling are contained in this section and allows for several different methods that with certain comments and cautions are acceptable. The sampling system design and the sample method selected will depend upon the composition, pressure temperature and hydrocarbon dewpoint of the sample at source conditions.

Ensure sample containers are clean prior to employing any of the methods.

The methods are:

a) evacuated container method,

b) reduced pressure method,

c) helium pop method,

d) floating piston container method,
e) glycol or water displacement method,

f) purging—fill and empty method,

g) purging—controlled rate method,

h) portable or online chromatograph method.

The evacuated container method, the reduced pressure method, and the helium pop method all require evacuating the sample container to 1 mm Hg (1/2 in. H₂O or 3.39 kPa abs) or less, absolute pressure.

The floating piston container method, the glycol displacement method, and the water displacement method are constant pressure methods, and are good choices when the pressure within the sample container should remain equal to the stream pressure during the sampling procedure.

The user should expect degradation in accuracy with any of the methods when the temperature of the source gas stream and/or all parts of the sampling system are not at or above the hydrocarbon dew point temperature.

Pressure gauges should be calibrated or compared to a certified pressure standard on a regular schedule to ensure accuracy. This is particularly important in reduced pressure sampling methods.

All sampling system components between the sample point valve and the sample container should be maintained above the hydrocarbon dew point temperature. To date, the most accurate determination of hydrocarbon dew point is the chilled mirror test. By definition, the gas-phase portion of the product flowing in a two-phase system is at the hydrocarbon dew point temperature. Calculated hydrocarbon dew points are only as accurate as the accuracy of the calculation method and the detail and accuracy of the analysis.

Any component of the sampling system that separates unwanted liquids from the sample stream should be operated at flowing line temperature and pressure.

Maintain the hydrocarbon dew point temperature in components downstream of the sample conditioning system, and where sample is to be purged.

For purposes of this publication, the hydrocarbon dew point temperature is considered one of the following: the flowing line temperature (at flowing line pressure), the value determined by a chilled mirror test (at flowing line pressure), or 30 °F (17 °C) above the calculated hydrocarbon dew point temperature. If the product flowing in the line is completely in the vapor phase the flowing line temperature is, of course, above the hydrocarbon dew point temperature for the line pressure. But if any droplets of liquid have condensed in the flowing product stream, the flowing line temperature is equal to the hydrocarbon dew point temperature for the line pressure. Therefore, if sample system components are maintained above flowing line temperature and at line pressure, the sampling system components will be above hydrocarbon dew point temperature (unless the line is 100% liquid full).

12.2 Evacuated Container Method

12.2.1 General

This method can be used when the vapor phase portion of the flowing stream is at or above the hydrocarbon dew point temperature.

The evacuated container method requires the container to have a vacuum of 1 mm Hg (1/2 in. H₂O or 3.39 kPa abs) or less absolute pressure or that the source pressure be at least 15 psig (103 kPa gauge).
When using the evacuated container method, the valves and fittings on the sample container should be in good condition and there should be no leaks in either the evacuated or pressurized states. Users are cautioned that most valve styles are not vacuum-tight. Therefore, it is recommended that the container be evacuated immediately prior to sample procurement.

This method has produced results to within ±0.14 % of the reference gas mixture heating value (HV) and density value when the sample gas temperature and all parts of the sampling system temperature are maintained according. The equipment arrangement is shown in Figure 8.

![Figure 8 Evacuated Container Method, Reduced Pressure Method, Helium Pop Method](image)

The procedure for sampling by the evacuated container method is as follows:

Evacuate the sample container to a pressure of 1 mm Hg or less using a container that has been previously evacuated and tested to hold 1 mm Hg pressure. Evacuation methods require that a vacuum gauge be permanently affixed to the sampling container to detect any leaks between the time of evacuation and the time of actual sampling in the field. Since many sample container valves are designed to hold pressure rather than vacuum, it is recommended that the vacuum be pulled immediately prior to sampling. Since standard vacuum gauges can only
be read to about 5% accuracy, it is further recommended that samples collected by evacuation methods be analyzed on the molecular sieve column to correctly determine the concentration of free nitrogen (see GPA Standard 2261).

To evacuate the container, attach the vacuum tubing to the outlet of the auxiliary valve. With the sample container inlet valve, sample container outlet valve and the auxiliary valve shut, start the vacuum pump. Open the auxiliary valve. Open the sample container outlet valve. When the vacuum gage indicates less than 1mm Hg pressure shut the sample container outlet valve.

Shut the auxiliary valve.

Open sample source valve (Valve 1) at sampling point and thoroughly blow out any accumulated material. Close sample valve (Valve 1) at sampling point.

Install sample container as shown in Figure 8 with the container in a vertical position. With all valves closed, open sample valve (Valve 1) at sampling point to fully open position.

Slowly purge the sample line with source gas to displace air by partially opening the purge valve until gas is slowly flowing. Close purge valve at sampling point.

While monitoring the vacuum gauge, slowly open the sample container Inlet valve, allowing the container pressure to increase to the source gas pressure. In some cases, condensation may be eliminated by sampling at a pressure less than the source pressure. (See Reduced Pressure Method.) It is possible to reduce pressure and produce retrograde condensation in the process. See discussion of the phase diagram.

Close the sample container Inlet valve and the sampling source valve (Valve 1). Open the purge valve to relieve pressure in the sample line. Depressurize appropriate portions of the sampling system.

Remove sample container and check for leaks by immersion of the valves in water or by the use of a commercial leak detection solution (applicable only for sample pressure greater than 15 psia). Plug or cap the valves using thread sealant.

NOTE: If the final pressure is below 5 psig the sample pressure should be increased to 15 psig in the laboratory by pressurizing with a gas that will not interfere with the analysis. If the sample is pressurized, the pressure existing immediately before and after pressurization shall be recorded.

12.3 Reduced Pressure Method

12.3.1 General

This method can be used when the vapor phase portion of the flowing stream is at or above the hydrocarbon dew point temperature and if the sample source pressure is at least 15 psig (103kPa gauge).

The reduced pressure method is similar to the evacuated container method except that instead of allowing the container to come up to line pressure, it is slowly filled to a point approximately one-third line pressure. There may be one or two objectives for reducing pressure: to reduce high pipeline pressures down to the pressure rating of the sampling equipment and/or to reduce the pressure enough to ensure that the sample is comfortably in the vapor phase. However, it is possible to reduce pressure and produce retrograde condensation in the process. This method is only applicable if the pressure reduction does not cross the phase boundary.

Since the hydrocarbon dew point temperature of the gas changes with pressure, filling the container to one-third line pressure does not guarantee that there will be no condensation. Consider a container at a temperature of 20 °F (-7 °C), used to sample an unknown stream at a pressure of 1500 psig (10.3 MPa). The reduced pressure method specifies filling the container to approximately 500 psig (3.4 MPa). However, the mixture may still condense at a pressure of 500 psig (3.4 MPa), and a temperature of 20 °F (-7 °C).
This method has produced results to within ±0.12 % of the reference gas mixture HV and density values when the sample gas temperature and all parts of the sampling system are maintained according section “General Discussion of Heating”

**SAFETY NOTE-** All sampling equipment should be rated for the highest expected sample pressure. Pressure reduction to sample system rated pressure should be accomplished with appropriately designed pressure regulators and pressure relief devices.

12.3.2 Procedure- Reduced Pressure Method

The reduced pressure method is frequently chosen when the sample pressure is greater than the rated pressure of the sample container. In such a situation, extreme caution should be used in the design of the sampling system. Refer to Note below. If a pressure-reducing regulator is used, the outlet valve of the regulator is considered to be Sample valve (Valve 1).

**NOTE-** Natural gas is often handled at high pressures. The sampling technician should be properly trained to work with compressed gasses. All sampling equipment should be rated equal to or higher than the highest expected pressure. Pressure reduction from pipeline to rated sample system pressure should be carried out with appropriately designed pressure regulators. Pressure relief devices should be used to protect from over pressure conditions.

It is possible to reduce pressure and produce retrograde condensation in the process. See Section for discussion of the Phase Diagram, Annex A.

The equipment arrangement is shown in Figure 8. The procedure for sampling by the reduced pressure method is as follows:

**Step 1:** Prior to sampling, evacuate the sample container to a pressure of 1 mm Hg or less. (Use a container that has been previously evacuated and tested to hold 1 mm Hg pressure) Evacuation methods require that a vacuum gauge be permanently affixed to the sampling container to detect any leaks between evacuation and actual sampling in the field. Since many sample container valves are designed to hold pressure rather than vacuum, it is recommended that the vacuum be pulled in the field immediately prior to sampling. Since standard vacuum gauges can only be read to about 5% accuracy, it is further recommended that samples collected by evacuation methods be analyzed on the molecular sieve column to correctly determine the concentration of free nitrogen (see GPA Standard 2261).

**Step 2:** To evacuate the container in the field, attach the vacuum tubing to the outlet of the auxiliary valve. With the sample container inlet valve, sample container outlet valve and the auxiliary valve shut, start the vacuum pump. Open the auxiliary valve. Open the sample container outlet valve. When the vacuum gauge indicates less than 1mm Hg pressure, shut the sample container outlet valve. Shut the auxiliary valve.

**Step 3:** At the sample point, open valve (Valve 1) at sampling point and thoroughly blow out any accumulated material. Close valve at sampling point.

**Step 4:** Install sample container as shown in Figure 8. The container should be in a vertical position. With all valves closed, open Sample Valve (Valve 1) at sampling point to fully open position.

**Step 5:** Slowly purge the sample line with source gas to displace air by partially opening the purge valve until gas is slowly flowing. Close purge valve at sampling point.
Step 6: While monitoring the vacuum/pressure gauge, slowly open the sample container inlet valve, allowing the container pressure to increase to the desired sampling pressure.

Step 7: Close the sample container inlet valve and the sampling valve (Valve 1). Open the purge valve to relieve pressure in the sample line. Close sample source valve. Depressurize appropriate portions of the sampling system.

Step 8: Remove the sample container and check for leaks by immersion of the valves in water or by the use of a commercial leak detection solution. Plug or cap the valves using thread sealant.

**NOTE**: It is not recommended that reduced pressure samples be taken when line pressures are below 100 psig.

### 12.4 Helium Pop Method

#### 12.4.1 General

The helium pop method requires preparing the container by evacuating the sample container to 1 mm Hg (\(1/2\) in. H2O or 3.39 kPa abs) or less absolute pressure. The helium pop method is similar to the evacuated container method except that a helium charge is used to keep the container free of air prior to sampling.

This method can be used when the vapor phase portion of the flowing stream is at or above the hydrocarbon dew point temperature. The gas source pressure should be greater than the helium pre-charge pressure. This method assumes that the chromatograph carrier gas is helium, that helium is not a component of interest and that helium does not interfere with any component of interest. Source pressure should be at least 15 psig (103 kPa gauge).

Use of the helium pop method will reduce the un-normalized total percent that is calculated during a gas chromatographic analysis. Therefore, the un-normalized total percent cannot be used as a gas chromatograph diagnostic when employing this method.

This method has produced results to within ±0.15 % of the reference gas mixture HV and density values when the sample gas temperature and all parts of the sampling system are maintained according to section “General Discussion of Heating”

#### 12.4.2 Procedure-Helium “Pop” Method

The equipment arrangement is shown in Figure 8. The procedure for sampling by the helium “pop” method is as follows:

Step 1: Prior to sampling, evacuate the container to a pressure of 1 mm Hg or less. (Use a container that has been previously evacuated and tested to hold this vacuum.)

Step 2: Prior to sampling, loosely connect the helium source to the auxiliary valve with the helium flowing out of the loose connection to purge the sample container auxiliary valve. Tighten the connection at the sample container auxiliary valve. Open the sample container auxiliary valve and allow helium to flow into the sample container until the pressure reaches approximately 5 psig. Close the sample container auxiliary valve. Close helium source valve. Remove container from helium source.

Step 3: At the sample point, open sample valve (Valve 1) and thoroughly blow out any accumulated material. Close valve at sampling point.

Step 4: Install the sample container as shown in Figure 8. The container should be in a vertical position. With all valves closed, open the sample valve (Valve 1) at sampling point to fully open position.
Step 5: Slowly purge the sample line with source gas to displace air by partially opening the purge valve until gas is slowly flowing. Close purge valve at sampling point.

Step 6: Slowly open the sample container inlet valve, allowing the container pressure to increase to the desired sampling pressure. In some cases, condensation may be eliminated by sampling at a pressure less than the source pressure. (See reduced pressure method, Section 12.3.) It is possible to reduce pressure and produce retrograde condensation in the process. See section for discussion of the phase diagram, Annex A.

Step 7: Close the sample container inlet valve and the sample valve (Valve 1). Open the purge valve to relieve pressure in the sample line. Depressurize appropriate portions of the sampling system.

Step 8: Remove the sample container and check for leaks by immersion of the valves in water or by the use of a commercial leak detection solution. Plug or cap the valves using thread sealant.

12.5 Floating Piston Container Method

12.5.1 General

This method can be used when the vapor phase portion of the flowing stream is at or above the hydrocarbon dew point temperature. The constant pressure container is designed to maintain the sample at pipeline pressure however the sample pressure should be sufficient to overcome the friction of the piston seals, allowing the piston to move. Source pressure should be at least 15 psig (103 kPa gauge).

The sample container should be reheated in the laboratory to a temperature necessary to ensure complete vaporization of any liquids in the container. It is also critically important to ensure that the container is properly cleaned before sampling, particularly if there is a suspicion that previous samples condensed within the container or if the container made contact with any liquid hydrocarbons such as slugs or compressor oil.

The piston sealing mechanism (normally O-rings or lip seals) requires that the piston totally separate the sampled natural gas from the precharge gas. The material used for the sealing mechanism and the lubricant used should not adsorb or in any other way distort any of the components in the natural gas mixture. Adsorption or absorption of hydrocarbon components can cause leakage and/or seal failure.

This method has produced results to within ±0.14% of the reference gas mixture HV and density values when the sample gas temperature and all parts of the sampling system are maintained according to section “General Discussion of Heating”

Floating piston containers should have the following features:

— They should be of sturdy construction and non-absorptive to the sample. Their design pressure rating should exceed the highest pressure available at the facility where they may be used.

— A piston position indicator, which is a method to detect leakage of precharge gas, should be provided.

— PTFE seals or equivalent, which requires no grease for sealing or smooth operation of the floating piston, should be used.

— Silicon grease or other piston lubricants that may absorb or contaminate the sample should not be used.

— The precharge side of the container should be charged to a pressure equal to or greater than line pressure. The piston should be flush with the inlet end cap. It is recommended that the precharge side be charged with an
inert gas that is different from the components in the stream being sampled. This will not affect analytical results should a leak occur.

Before the sampling operation is begun, the associated sample inlet piping should be purged through the end cap purge valve on the inlet side of the piston container. If the container is not equipped with a purge valve, a “purge-valve tee” arrangement should be installed to allow purging the inlet piping and valving. Care should be taken to avoid condensation and contamination during the purge procedure.

12.5.2 Procedure- Floating Piston Container Method

12.5.2.1 General

The lubricant on the floating piston container moving parts should be applied as lightly as possible and no component of the sample can be soluble in the lubricant. Refer to Annex I for cleaning recommendations.

It is desirable to use an inert charge gas for the floating piston container, which is not present in the sample so that a leak in the container itself can be easily detected during the analysis. Natural gas from the sample source can be used in an emergency. Charge the inert gas side of the container with pressure equal to or greater than the line pressure.

12.5.2.2 Sampling with a piston container equipped with a visual indicator

Step 1: Open the sample valve (Valve 1) and thoroughly blow out any accumulated material. Close the sample valve (Valve 1).

Step 2: Install the sample container as shown in Figure 9.

Step 3: Open the sample valve (Valve 1) and slowly purge the sample line with source gas by partially opening the purge valve until gas is slowly flowing out of the purge valve. Close the purge valve.

Step 4: Slowly open the sample container inlet valve to fully open position and observe pressure on Gauge M. Adjust pressure on Gauge N to equal pressure on Gauge M by releasing inert gas through the sample container inert gas valve.

Step 5: Partially open the sample container inert gas valve to allow the inert gas to slowly vent to atmosphere. Do not allow the pressure as indicated by Gauge M to drop below sampling pressure. Continue operation until the indicator designates the container to be approximately 80 % full.

Step 6: Close the sample container inert gas valve, the sample container inlet valve and the sample valve (Valve 1).

Step 7: Relieve the pressure on the sample transfer line using the purge valve. Depressurize appropriate portions of the sampling system.

Step 8: Remove the sample container and check the sample gauge, sample container inlet valve and sample container inert gas valve for leaks. Plug or cap the valves using thread sealant.

NOTE- Some pressure is required to move the piston inside these containers. Therefore, this method may not be appropriate in the sampling of gases of very low pressure.
12.5.2.3 Sampling with a piston container not equipped with a visual indicator

Step 1: Open the sample valve (valve 1) and thoroughly blow out any accumulated material. Close sample valve (valve 1).

Step 2: Install the sample container as shown in Figure 9.

Step 3: Open the sample valve (valve 1) and slowly purge the sample line with source gas by partially opening the purge valve until gas is slowly flowing out of the purge valve. Close the purge valve.

Step 4: Slowly open the sample container inlet valve to fully open position and observe pressure on gauge M.
Step 5: Adjust pressure on gauge N to equal pressure on gauge M by releasing inert gas through the sample container inert gas valve.

Step 6: Partially open the sample container inert gas valve to allow the inert gas to slowly vent to atmosphere. Do not allow pressure gauge N to drop below sampling pressure. Continue operation until the container is approximately 80 % full.

Step 7: Close the sample container inert gas valve, the sample container inlet valve and the sample valve (valve 1).

Step 8: Relieve the pressure on the sample transfer line using the purge valve. Depressurize appropriate portions of the sampling system.

Step 9: Remove sample container and check sample gauge, sample container inlet valve and sample container inert gas valve for leaks. Plug or cap the valves using thread sealant.

**NOTE:** Some pressure is required to move the piston inside these containers. Therefore, this method may not be appropriate in the sampling of gases of very low pressure.

### 12.6 Glycol or Water Displacement Method

#### 12.6.1 General

This method can be used when the vapor phase portion of the flowing stream is at or above the hydrocarbon dew point temperature. The user is cautioned to remove all glycol or water during the sampling process. The glycol or water, if left in the sample container, may absorb or desorb sample components such as CO2 or H2S, depending on the water quality or contact time. Using distilled water will prevent desorption but not absorption of CO2 and other components. Glycol or water can also cause damage to the analytical equipment. This method should not be used when water or other glycol-soluble components are included in the components of interest. When the ambient temperature is below 32 °F (0 °C), glycol should be the displacement medium. When the ambient temperature is above 32 °F (0 °C), glycol typically in a 50/50 water mixture, or water may be used as the displacement medium. Source pressure should be at least 15 psig (103 kPa gauge). This method should not be used when sampling for water content determination.

The water displacement method has produced results to within ±0.13 % of the reference gas mixture HV and density values when the sample gas temperature and all parts of the sampling system are maintained according to section “General Discussion of Heating”.

The glycol determination method has produced results to within ±0.10 % of the reference gas mixture HV and density values when the sample gas temperature and all parts of the sampling system are maintained according to section “General Discussion of Heating”.

#### 12.6.2 Procedure-Glycol or Water Displacement Method

The equipment arrangement is shown in Figure 10.
The procedure for sampling by the Glycol or Water Displacement Method is as follows:

Step 1: A sample container filled with clean glycol or water and a vessel to measure the displaced liquid are required for this method. Water should have a pH between 5.0 and 7.0. Water with a pH greater than 7.0 should be acidified to bring the pH within the proper range. (0.1N H₂S₀₄ is a satisfactory acid solution.) The source gas should displace all glycol or water during sampling.

Step 2: Open the sample valve (Valve 1) at sampling point and thoroughly blow out any accumulated material. Close the sample valve (Valve 1) at sampling point.

Step 3: Install the sample container as shown in Figure 10. The container should be in a vertical position. With all valves closed, open the sample valve (Valve 1) at sampling point to fully open position. Slowly purge the sample line with source gas to displace air by partially opening the purge valve until gas is slowly flowing. Close the purge valve at the sampling point.
Step 4: Slowly open the sample container inlet valve to fully open position.

Step 5: Slowly open the sample container outlet valve to allow a slow discharge of the glycol or water displacement liquid into the measuring vessel until gas is seen or heard. Immediately close the sample container outlet valve.

Step 6: Close the sample container inlet valve and the sample valve (Valve 1). Open the purge valve to relieve pressure in the sample line. Depressurize appropriate portions of the sampling system.

Step 7: Remove the sample container and check for leaks by immersion of the valves in water or by the use of a commercial leak detection solution. Plug or cap the valves using thread sealant.

**NOTE:** All of the displacement liquids should be removed from the container during sampling to prevent noticeable change in gas composition.

**NOTE:** Displaced water and glycol should be collected and handled in accordance with all relevant environmental and safety regulations.

### 12.7 Purging—Fill and Empty Method

#### 12.7.1 General

This method can be used when the flowing stream is above the hydrocarbon dew point temperature. The temperature of the sample equipment downstream of the sample valve (Valve 1) should be maintained above the expected hydrocarbon dew point throughout the sampling process. It is necessary to use an extension tube ("pigtail") with a flow control drilled plug or valve when using this method. It is important to adhere to the recommended number of purge cycles (See Table 2) to ensure that a representative sample is secured.

The sample cylinder can be attached to the sample probe in one of two ways. It can be direct mounted (See Figure 1a) or tube mounted (See Figure 1b). In a direct mount, the sample cylinder is attached directly to the valve on the sample probe. This reduces the amount of tubing and fittings between the probe and the sample cylinder, reducing the potential for contamination and leaks. However, if liquids are present, they can build-up within the cylinder. In a tube mount, the tubing between the probe and the sample cylinder orients the cylinder so that the sample flows downward and out through pigtails, reducing the chance of sample distortion. Source pressure should be at least 15 psig (103 kPa gauge).

### Table 2- Fill and Empty Cycles

<table>
<thead>
<tr>
<th>Maximum Gas Pressure in Container, psig (kPa guage)</th>
<th>Number of Fill and Empty Cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>15-29 (100-200)</td>
<td>13</td>
</tr>
<tr>
<td>30-59 (200-405)</td>
<td>8</td>
</tr>
<tr>
<td>60-89 (405-615)</td>
<td>6</td>
</tr>
<tr>
<td>90-149 (615-1025)</td>
<td>5</td>
</tr>
<tr>
<td>150-500 (1025-3450)</td>
<td>4</td>
</tr>
<tr>
<td>&gt;500 (&gt;3450)</td>
<td>3</td>
</tr>
</tbody>
</table>

# of Purge Cycles = \log_{10} x y

Where:
- \( x \) = atmospheric pressure + fill pressure (psia)
- \( y \) = maximum allowable mole fraction of contaminants
The purging—fill and empty method requires several cycles of filling and emptying the sample container in order to remove residual impurities. Table 2 shows the number of fill and empty cycles required to sufficiently remove residual impurities from the container.

This method has produced results to within ±0.12% of the reference gas mixture HV and density values when the sample gas temperature and all parts of the sampling system are maintained according to section 6.12 “General Discussion of Heating”.

This method requires that:

— the container is coupled to the sample point as shown in Figure 11
— a “pigtail” with a flow restriction at the end is attached to the outlet of the container as shown in Figure 11. The key consideration is that the cooling produced by the Joule–Thomson effect should be located at the end of the “pigtail”;
— the container is purged of residual impurities using the correct number of fill and empty cycles (Table 2)

![Figure 11- Purging-Fill and Empty, Purging-Controlled Rate Methods](image)

12.7.2 Procedure- Purging - Fill and Empty Method

The equipment arrangement is shown in Figure 11 and 12.
To minimize the potential for ambient cooling, the tubing and purge valve section between the sample point outlet valve (Valve 1) and the sample container inlet valve should be kept as short as possible. It is necessary to connect an extension tube ("pigtail") (refer to Section "extension tube") with a valve or orifice restriction on the discharge end to the outlet valve of the container. This arrangement pushes the temperature drop associated with the Joule-Thomson Effect to the end of the extension tube ("pigtail") and eliminates the possibility of heavy hydrocarbon condensation in the sample container outlet valve. To facilitate rapid blow-down for the Purging-Fill and Empty Method, a flow-regulating valve may be used to replace the flow plug. The internal diameter of the flow-regulating valve should be the smallest diameter of all the sampling system components.

The temperature of the sample equipment downstream of the sample valve (Valve 1) should be maintained above the expected hydrocarbon dew point throughout the sampling process. If the sample container is initially pre-heated to line temperature, studies have shown that the heat of compression generated during the fill and empty cycles will keep the sample container above the expected hydrocarbon dew point temperature.

Step 1: Open the valve (Valve 1) at the sampling point and thoroughly blow out any accumulated material. Close the valve at the sampling point. The purge time should be long enough to blow out contaminants but not so long as to chill the sample point valve.

Step 2: Install the sample container as shown in Figure 11 or 12. The container should be in a vertical position. With all valves closed, open the sample valve (Valve 1) at the sampling point to fully open position.
Step 3: Open the purge valve at the sampling point and gently blow out any accumulated material. Close the purge valve.

Step 4: Slowly open the sample container inlet valve to pressure up the sample container.

**NOTE**- There is a theoretical possibility for auto-ignition of natural gas and air mixtures in gas sample containers if improperly filled (rapidly pressurized). Rapid opening valves should not be used in the sampling system. The only exception is that the outlet of the sample probe may have a ball valve. Care should be taken to avoid rapidly opening the sample probe outlet valve to an open container that is filled with air. The procedures outlined in this method are designed, through valve operation sequences, to prevent rapid filling of the container.

Step 5: Open the sample container outlet valve. Open the extension tube ("pigtail") line valve. Purge the line slowly with gas to displace air. (Valves 1, sample container inlet valve, the sample container outlet valve and the extension tube ("pigtail") line valve should be fully opened. The purge valve should be shut.)

Step 6: Close the extension tube ("pigtail") line valve and allow the pressure to build up rapidly to sample supply pressure.

Step 7: Close the sample container inlet valve. Open the extension tube ("pigtail") line valve and vent the container through the extension tube ("pigtail") line valve to almost atmospheric pressure. Close the extension tube ("pigtail") line valve.

Step 8: Open the sample container inlet valve and allow the pressure to build up rapidly to sample supply pressure.

Step 9: Repeat the purge steps from above to comply with Table 2 which gives the minimum number of cycles required to effectively purge the container of the original gas in the container.

Step 10: Close the sample valves in the following order: extension tube ("pigtail") line valve; sample container outlet valve; sample container inlet valve and sampling valve (Valve 1).

Step 11: Slowly open the extension tube ("pigtail") line valve. Observe for trace of liquid in the discharge of the extension tube ("pigtail") line valve. If liquid appears at the extension tube ("pigtail") line valve, discard the sample and reselect a sample method as outlined in Section 12 “Sampling Methods”. Also review Section 6.12 “Heating Requirements”.

Step 12: Open the purge valve slowly to relieve pressure on the sample connection. Depressurize appropriate portions of the sampling system.

Step 13: Remove the sample container and check for leaks by immersion of the valves in water or by the use of a commercial leak detection solution.

Step 14: Plug or cap the valves using thread sealant.

**12.8 Purging—Controlled Rate Method**

**12.8.1 General**

This method employs a continuous purge and can be used only when the vapor phase portion of the flowing stream is above the hydrocarbon dew point. Source pressure should be sufficient to produce critical flow conditions in flow plug (pressure of 15 psig (103 kPa gauge) or greater and flow tube exhaust to the atmosphere). Extreme care should be
used to prevent condensation during the sampling process. It is necessary to use an extension tube ("pigtail") with a drilled plug when using this method. It is important to adhere to the recommended purge time (See Charts 1, 2, 3 or 4) to ensure that a representative sample is secured.

NOTE This method has produced results to within ±0.18% of the reference gas mixture HV and density values when the sample gas temperature and all parts of the sampling system are maintained according to section "General Discussion of Heating"
12.8.2 Procedure- Purging - Controlled Rate Method

The equipment arrangement is shown in Figure 11 or 12. Source pressure should be sufficient to produce critical flow conditions in flow plug (pressure of 15 psig or greater and flow tube exhaust to the atmosphere). Deviations from the flow curves will occur when the gas specific gravity is other than 0.600 and the flowing temperature is not 60 °F, however, the variation will likely be less than 10%. For a flow plug bore diameter of 0.125", purge time should be 1 min for every 100 cm$^2$ of sample container volume. The procedure for sampling by this method is as follows:

Step 1: Open the valve (Valve 1) at the sampling point and thoroughly blow out any accumulated material. Close the valve at the sampling point. The Purge time should be long enough to blow out contaminants but not so long as to chill the sample point valve.

Step 2: Install the sample container as shown in 11 or 12. The container should be in a vertical position. With all valves dosed, open the valve (Valve 1) at the sampling point to the fully open position.

Step 3: Open the purge valve at the sampling point and gently blow out any accumulated material. Close the purge valve.
Step 4: Slowly open the sample container inlet valve fully.

Step 5: Slowly open the sample container outlet valve fully.

Step 6: Slowly open the extension tube (“pigtail”) line valve fully.

Step 7: Assuming a 0.125" diameter hole in the flow plug, flow in this manner 1 min for each 100 cm² of the sample container volume. Charts 1, 2, 3 and 4 show flow times for various hole diameters and various sample container sizes.

The calculation for minimum flow time is:

\[
Purge Time in Seconds = \frac{Cylinder Volume in cc}{10,800 \times (Hole Diameter in inches)^2}
\]

Close the sample valves in the following order: extension tube (“pigtail”) line valve; sample container outlet valve; sample container inlet valve and sampling valve (Valve 1).

Step 8: Slowly open the extension tube (“pigtail”) line valve. Observe for trace of liquid in the discharge of the extension tube (“pigtail”) line valve. If liquid appears at the extension tube (“pigtail”) line valve, discard the sample and select a sample method for a flowing gas stream that is at the hydrocarbon dew point as outlined in Section 12. Also review Section 6, “Heating Requirements”.

Step 9: Open purge valve slowly to relieve pressure on the sample connection. Depressurize appropriate portions of the sampling system.

Step 10: Remove the sample container and check for leaks by immersion of the valves in water or by the use of a commercial leak detection solution. Plug or cap the valves using thread sealant.

12.9 Vacuum-gathering System Method

12.9.1 General

In rich, low-pressure, or vacuum-gathering systems, the use of a vacuum pump is recommended. One method involves a pump drawing gas from the sample point and discharging into the sample system (see Figure 13).
Figure 13-Vacuum Gathering System Method

An alternate method, not evaluated in the research program (see Figure 14), involves drawing the sample through a helium-filled sample container with the pump.
12.9.2 Procedure – Vacuum-gathering System Method

With either method, before the sample is collected, both the oxygen content and the relative density of the stream should be measured using a portable oxygen analyzer and a portable gravitometer to ensure the sample is representative of earlier samples and free of air contamination due to leaks. For the method shown in Figures 13 and 14 once the oxygen and gravity values are recorded and accepted, the sample is collected using any of the following methods:
— Fill and Empty
— Evacuated Container
— Helium Pop
— Glycol / Water Displacement

Pressure within the sample container is maintained at less than 20 psig (13.8 kPa). Without sufficient heat input, increased sample pressure may cause condensation to occur. Sample containers larger than 300 cm³ (18 in³) may be required to contain enough material to analyze. It is extremely important that samples from such streams be heated in accordance with the recommendations in Section 6.12 if analytical results are to be repeatable and reproducible.

Samples of compositions characteristic of vacuum-gathering systems (very high HV content) demonstrate a greater degree of uncertainty in the analytical results due to the susceptibility of the heavy ends to condense.

12.10 Portable and On-Line Gas Chromatograph Method (see GPA 2261)

12.10.1 General

The portable and on-line gas chromatograph sampling method can be used when the vapor phase portion of the flowing stream is at or above the hydrocarbon dew point temperature. It may be necessary to use a regulator to reduce line pressure to the required pressure for the portable or on-line gas chromatograph.

Pressure reduction through the regulator will cause a drop in temperature, which may cause the gas to drop below the hydrocarbon dew point. To counteract the effects of the Joule-Thomson Effect, ambient cooling or retrograde condensation, it may be necessary to heat trace the regulator and sample tubing. See Section 6.12 General Discussion of Heating. Source pressure should be at least 15 psig (103 kPa gauge).

Refer to GPA 2261 for information relative to the use of a portable chromatograph.

12.10.2 Sample Transfer Line

12.10.2.1 Material

A sample transfer line should be non-reactive to the sample. It should be rated for the maximum expected pressure and should retain its strength at the maximum or minimum expected temperature. Stainless steel (304 or 316) and PTFE or PFA or similar) have been proven to be non-reactive to natural gas components. Stainless steel is preferred for pressures over 1000 psig, or for gases containing hydrogen sulfide (consider silica or other non-reactive material-lined stainless steel tubing for hydrogen sulfide applications, as stainless steel is known to adsorb H₂S).

NOTE- PTFE is an appropriate alternative for moisture analyses.

12.10.2.2 Length

The sample transfer line should be as short as possible. Inches (centimeters) are preferred to feet (meters). Short lines reduce the heat transfer with ambient surroundings. If ambient temperatures are lower than the temperature of the system being sampled, cooling of the sample tubing can cause condensation of the heavier (higher boiling point temperature) components. These condensed components can then drop out or adhere to the wall, causing the sample to be lighter (fewer heavy components, lower heating value). Or the accumulated droplets can be swept into the sample container, causing the sample to be heavier (higher heating value) than the product to be sampled.
For the portable or on-line gas chromatograph method, sample transport lag time calculations are helpful in determining appropriate sample bypass purge rate. The below formula will allow the calculation of the actual sample transport time.

\[
 t = \frac{VL}{F_s} \left[ \frac{P + 15}{15} \right] \times \left[ \frac{530}{T + 460} \right] \times \left[ \frac{1}{Z_p} \right]
\]

Where:

- \( t \) = Purge Time
- \( L \) = Line Length, ft
- \( V \) = Tubing Volume, cc/ft
- \( F_s \) = Standard Flow, cc/min
- \( P \) = Actual Pressure, PSIG
- \( T \) = Actual Temperature, °F
- \( Z_p \) = Compressibility at P Pressure

* This formula was taken from Appendix F; pp 579 and 580. “Process Analyzer: Sample Conditioning System Technology” by Robert E. Sherman; Wiley Interscience, and modified for Gas Compressibility by ABB Totalflow Project Engineering.

### 12.10.2.3 Diameter

The diameter should be as small as possible while ensuring adequate flow through the system.

For spot sampling, the transfer line should have a minimum diameter of 1/4 in (6.35 mm). To ensure that refrigeration associated with the Joule-Thomson Effect occurs some distance downstream of the sample container, the smallest diameter component in the spot sample system should be the orifice in the drilled plug at the end of the extension tube (“pigtail”) attached to the outlet of the sample container. The drilled plug can be replaced with a flow control valve provided that the internal diameter of the flow control valve is smaller than any other component in the sampling system.

For the portable and on-line gas chromatograph method, the sample transfer line would generally have a diameter of 1/16 in (1.59 mm) to ¼ in (6.35 mm). For the portable or on-line gas chromatograph method, sample transport lag time calculations are helpful in determining appropriate sample line diameter.

Sample transfer lines should be maintained above the hydrocarbon dew point.

For very short connections between the sample point and the sample container, the sample transfer line can be insulated without supplemental heating. Care should be taken to ensure that this short transfer line is at the same temperature as the sample source prior to transferring sample to the sample container.

### 12.11 Use of Thermal Isolation and Throttling Devices

#### 12.11.1 General

The purpose of thermal isolation and throttling devices is to prevent cooling of the sample container during the purging process. (Refer to Figure 12) Throttling the sample gas downstream of the container outlet valve reduces both the pressure drop across the sample container valves and the resulting Joule–Thomson cooling effects caused by upstream restrictions. Thermal isolation of the throttling device from the container prevents cooling of the container.

The smallest diameter portion of the sample system will be the point of maximum pressure drop. This pressure drop causes reduction in temperature due to the Joule-Thomson Effect. Extension tubes ("pigtails") are used to thermally isolate the outlet valve of sample containers from this cooling effect during the purging process.
To ensure that cooling from flow regulation occurs at the outlet end of the pigtail, a flow regulating valve or flow regulating plug should be installed at the end of the pigtail. The internal diameter of the flow regulating valve should be the smallest diameter of all the sampling system components. The hole in the plugs should be the smallest diameter part of the sample system. Details of the flow plug are shown in Figure 15. To achieve the optimum heat-of-compression results in the Purging-Fill and Empty Method.

1/4 inch NPT Hex Head Pipe Plug 316 Stainless Steel.

Figure 15 – Flow Plug 1/8 port

When performing the fill-and-empty spot sampling procedure, use an extension tube ("pigtail") of 1/4 in. (6.35 mm) ID and at least 36 in. (91.4 cm) in length extending from the sample container outlet valve to the purge valve/throttling assembly on the end of the pigtail (Figure 12). The pigtail is intended to provide thermal isolation between the throttling device and the sample container outlet valve. This piece of tubing is typically 1/4 in. (6.35 mm)" in diameter and at least 36" in length. If it is coiled, the user is cautioned to leave enough air gaps between coils to prevent thermal coupling of the coils.

The throttling device utilized shall have a flow coefficient (Cv) ranging from 0.09 to 0.53. This corresponds to orifice diameters ranging from 1/16 in. to 1/8 in. (1.59 mm to 3.75 mm). A standard 1/16 in. to 1/8 in. port (1.59 mm to 3.75 mm port) multi-turn sample valve has also been shown to work well in this application. Other devices or combinations of devices are acceptable if they provide temperature isolation and throttling of the sample gas flow equal to or exceeding that of the “pigtail” and throttling devices described above.

Increasing the flow restriction will lengthen the purge time. In ambient temperature conditions at or below the hydrocarbon dew point of the flowing stream, the purge time may be lengthened to a point where the ambient air cools the sample container more than the fill and empty purging process can warm it. Insufficient throttling may cause excessive cooling of the sample gas at points of restriction upstream of the throttling device, allowing the gas sample to cool below its hydrocarbon dew point temperature. Allowing the gas sample to cool below its hydrocarbon dew point temperature may cause it to become non representative.
13 Automatic Sampling

13.1 Composite Samplers

13.1.1 General

Composite samples are automatically taken over an extended period of time, generally within a month with the sampling rate proportional to flow rate or time. There are several composite samplers commercially available. For streams with variable flow rate and composition, a flow-proportional sampler is recommended. Refer to Figure 1a and 1b above.

Condensation in the sample system should be avoided. Tests conducted under actual field operating conditions have shown that composite samplers cannot be reasonably expected to provide representative samples when exposed to ambient conditions below the sample gas hydrocarbon dew point. The gas being sampled should be maintained above the hydrocarbon dew point throughout the sampling process to ensure the sample remains in the gas phase from the sample point through the collection vessel. The application of supplemental heat to the sampling system may be necessary to counteract possible condensation caused by the Joule-Thomson Effect or fractionation of the sample when the temperature of the sampling apparatus falls below the hydrocarbon dew point. It is imperative that the gas being sampled be maintained above the hydrocarbon dew point throughout the sampling process.

13.1.2 Displacement Samplers

A positive displacement pump extracts a sample at line pressure and discharges it into a sample container during the sampling period.

13.1.3 System Considerations

The sample system should consist of one sample probe, one composite sampler, and one sample container, and the line between the sampling device and the collection container should be of minimum length. Heat and insulation may be required to avoid condensation. If the sample system does not provide a continuous flow of sample, the sampler should purge itself prior to pumping a sample increment into the collection container.

When using displacement samplers, either constant volume or constant pressure containers may be used. When using regulator samplers, constant volume containers are required.

13.2 Continuous Sampling Systems for On-line Analyzers

For on-line analyzers, such as chromatographs and gravimetric analyzers, the sample system will consist of components to extract, condition and deliver a representative sample of natural gas to the analyzer. The sample delivery system should not interfere with the integrity of the primary metering system. To avoid the possibility of system interference, it is generally recommended that on-line sampling systems be dedicated to a single device. See GPA Standard 2261 for more guidance.

14 Sampling Intervals

14.1 General Considerations

A sampling system shall provide a sample representative of the gas flowing in the pipeline. Since pipeline flow rates and compositions may vary with time, a sampling interval, time or flow proportional, should be carefully chosen so that the collected sample reflects these variations.
14.2 Composite Sample Intervals

In choosing the method to be used in pacing the sampler, the sample source stream is the primary concern. Flow proportional composite sampling systems are most likely to produce a representative sample. If the stream has a constant composition or flow rate, a time pacing mechanism may be used. Provisions in time-based systems shall be made to stop sampling when there is no flow.

14.3 Spot Sampling Intervals

Generally, gas pipeline composition will have daily, monthly, semi-annual, and seasonal variations. Compositional variations will also occur because of surface equipment and gas reservoir changes. All of these environmental and operational considerations should be taken into account when selecting a sampling interval for a spot sample. Spot samples will produce accurate and representative compositions only if the product composition is stable within the accounting time frame.

15 Labeling, Handling, and Transportation of Containers

15.1 Labeling

Labels or tags should be completed and associated to each sample container with the following information.
— Container ID (if applicable)
— Container contents: natural gas
— Sample source
— Pressure and temperature of the sampled source stream near the sample point
— Date and time of collection
— Field technician name

Additional information may be required, such as the following.
— Hydrocarbon dew point
— Sample type (spot or composite)
— Sample collection method
— Ambient temperature
— Water dew point and/or concentration
— Flow rate
— Relative density
— Oxygen concentration
— CO₂ concentration
— H₂S concentration

Labels or tags containers should not interfere with the utilization of the container. Figure 16 gives an example of typical label information.

US DOT CFR 49 includes specific requirements for labeling.
15.2 Handling and Transportation of Containers

Sample containers containing natural gas samples should be handled carefully due to their pressure, flammability, and/or contents.

The following actions shall be performed in conjunction with handling and transporting a sample.
- Check valves and fittings for leaks.
- Inspect, repair, or replace valves as required.
- Plug or cap sample container inlet and outlet valves prior to transportation container
- Avoid over-tightening valves; hand tightening of valves is sufficient.
- Protect sample containers from damage, by properly restraining the containers in strong crates or cases.
- Properly restrain crates and cases during transport.

Consider that depressurizing sample containers may produce low temperatures, high fluid velocities, and hazardous vapors.
It is recommended that all rigid sample containers incorporate an overpressure relief device, approved by the appropriate regulatory agency.

15.3 Sample Handling in the Laboratory

All laboratories shall meet the requirements of the GPA or other recognized standards organizations for the type of gas analysis configuration being used. All analyses shall be performed in accordance with procedures set forth in GPA publications or other accepted industry standards. (See Annex E.)

15.4 Sample Receipt and Storage

Upon arrival at the laboratory, each sample container should be logged into the laboratory’s sample tracking system to ensure the sample information and procedures performed upon it are documented from receipt through the cleaning of the sample container.

Each sample container shall be inspected for leaks upon arrival at the laboratory. If the sample appears to have been distorted or depleted due to leakage, another sample should be secured, if practicable. If another sample cannot be secured, applicable company procedures for a failed sample should be followed.

Sample containers should be stored in an environmentally controlled location. Transparent or semi-transparent sample containers should be stored in a dark location to guard against sample distortion due to any potential photosensitivity of the compounds contained within. Any samples suspected of containing reactive materials shall be stored in an inert environment.

15.5 Sample Handling Prior to Analysis

Sample containers shall be heated to a minimum of 30°F above the sampling source temperature or the hydrocarbon dewpoint of the sample, if known. Containers shall be held at that temperature for a minimum of 2 hours prior to sample analysis and maintained at that temperature until the sample has been introduced to the analyzer.

15.6 Sample Disposal and Container Cleaning

After obtaining acceptable sample analyses, sample containers shall be emptied in accordance with all applicable safety practices and regulatory guidelines.

Sample containers shall be cleaned and inspected for return to use in accordance with Annex I - Cleaning.

16 Auto-ignition of Natural Gas

There is a theoretical possibility for auto-ignition of natural gas and air mixtures in gas sample containers if improperly filled (rapidly pressurized) and the user is warned to exercise due caution.

If natural gas is introduced into the sample container at sonic velocity (the speed of sound), it is theoretically possible that the shock wave produced by the gas entering the container will act like a piston. The air–gas mixture could compress so rapidly that the mixture could reach its auto-ignition temperature. If the mixture is within the range of air–gas ratios that support combustion, a fire inside the container or an explosion could occur. For such an event to occur under these circumstances, the inlet valve would have to have a relatively large opening and be opened very quickly, as might occur with a quarter-turn, full-port valve.

To mitigate the theoretical possibility of this occurring, either the air/oxygen should be removed from the container or the maximum velocity of the gas entering the container should be below sonic velocity. This should be accomplished in a manner that does not introduce sample distortion.
Annex A
(Informative)

The Phase Diagram

A.1 Phase Changes in General

The importance of avoiding condensation during gas sampling is discussed throughout this standard because phase changes have a significant impact on the accuracy of a gas sample. When a hydrocarbon mixture undergoes a phase change process during sampling, the composition of the collected sample will not be the same as the composition of the flowing gas stream. The errors in composition resulting from a phase change can be large.

The phase diagram is a tool for modeling the phase behavior of a hydrocarbon mixture. The phase diagram illustrates the change in dew point and bubble point temperatures with changes in the gas pressure.

Since pressure changes are impossible to avoid during the sampling process (i.e., getting a sample of gas from the source, to the analyzer), an understanding of the gas mixture’s phase behavior provides guidance in the design and application of sampling systems and sampling methods.

For the purposes of this discussion, only liquid and gas phases are considered.

A.2 Single-component Phase Behavior

Consider the piston-container device shown in Figure A.1 (below) and filled with a single component (e.g., CO₂, N₂, He) in the gaseous phase. The pressure, temperature, and volume of the substance are represented by the letter A. If the substance is compressed isothermally (i.e., at constant temperature, T₁), there will be a decrease in the container volume. This decrease in volume and the associated increase in pressure will continue until liquid begins to condense. The pressure at which condensation begins is called the dew point, and is shown in Figure A.1 as point B.

Once condensation begins, additional decreases in volume produce more and more condensation (C and D) until only an infinitesimal amount of gas remains in the mixture. This pressure is called the bubble point pressure. In Figure A.1, it is point E. For a single component, the dew point pressure is equal to the bubble point pressure when the temperature is held constant. The substance is entirely in the liquid phase at volumes below the bubble point volume.

If this process is repeated for a temperature, T₂ greater than T₁, similar pressure–volume behavior occurs. This can be repeated at higher and higher temperatures until no distinct phase change occurs. This happens at the critical temperature. The pressure associated with the critical temperature is called the critical pressure. The critical point (CP) is the intersection of the critical temperature and critical pressure. Gas and liquid cannot coexist above the critical point of a single component.

When connected, the dew points, bubble points, and critical point form a region called the two-phase region. In this region, liquid and gas coexist in relative quantities ranging from just under 100 % gases, to just fewer than 100 % liquid.

If the dew point and bubble point pressures and temperatures are plotted on a pressure–temperature diagram, the result is a single line known as the P–T diagram, phase diagram, or vapor pressure curve. Figure A.1 shows a typical vapor pressure curve for a single component. The upper section corresponds to the liquid phase and the lower section corresponds to the gaseous phase. Gas and liquid will coexist at the pressure/temperature
points on the curve. The vapor pressure curve for a single component does not show the relative amounts of each phase.

### A.3 Mixture Phase Behavior

Now consider the piston-container device filled with a mixture of components, such as hydrocarbons. If the mixture is compressed isothermally, as in the previous example, the mixture will go from a gaseous phase to a mixture of gas and liquid, then to liquid, as shown on the T1 isotherm of Figure A.2. This can be repeated at progressively higher temperatures until the critical temperature is reached. The line connecting the dew points, bubble points, and the critical point forms the two-phase region.

The general phase behavior of a mixture, such as a hydrocarbon mixture, is similar to that of a single component, with two important exceptions. First, the dew point pressure does not equal the bubble point pressure at a given temperature. Second, gas and liquid can coexist at pressures and temperatures above the critical point of a mixture.

If the dew point and bubble point pressures and temperatures are plotted on a P–T diagram as shown in Figure A.2, the result is the mixture phase diagram. The region enclosed by the bubble points and dew points is the two-phase region, also known as the phase envelope.

Natural gas engineering is primarily concerned with mixture temperatures that are above the critical temperature.

### A.4 Retrograde Condensation and Vaporization

The vapor pressure curve for a single component was discussed earlier and is shown in Figure A.1. This curve represents the pressures and temperatures where two phases of a single component coexist. At a given temperature, the phase change process for a single component occurs at a constant pressure.

Mixture phase behavior is different than single component phase behavior. There is a pressure change during the phase change process. This pressure change appears on the phase diagram (P–T curve) as two saturation pressures for a given temperature (Figure A.2).

Focusing on the section of the phase diagram with temperatures above the critical temperature (Figure A.3), one can see that during an isothermal pressure drop, from point A to point E, the mixture starts completely in the gas phase, then begins to condense as it reaches the point labeled as the “upper dew point.” This “retrograde” condensation is counter to the behavior that occurs with a single component.

As the pressure continues to drop, more of the mixture condenses, until the percent of condensed liquid in the mixture reaches a maximum (determined by the composition of the original mixture). Once the maximum is reached, further pressure reduction causes a vaporization of the liquid until the “lower dew point” is reached. The mixture is entirely in the gas phase at pressures below the lower dew point pressure.

The opposite will occur during an isothermal pressure increase. It may also occur when gas from a vacuum-gathering system is compressed into the sample container.

Retrograde phase changes can also occur when the temperature is changed at pressures above the critical pressure and within the phase envelope.

### A.5 Natural Gas Mixture Phase Diagrams

Figure A.4 shows a phase diagram for a typical natural gas mixture. Several sections of the curve are labeled. The composition of the hydrocarbon mixture being modeled is also shown.
The line A–B is the section of the phase diagram known as the bubble point curve. When the pressure is lowered isothermally to the bubble point, an infinitesimal amount of gas begins to evolve. As the pressure is reduced further, more and more gas is liberated from the mixture, increasing the total concentration of gas in the two-phase mixture.

The line B–E is the dew point curve. This section of the phase diagram represents the pressures and temperatures associated with the condensation of an infinitesimal amount of liquid from the gas mixture.

The line C–D is sometimes referred to as the retrograde dew point line. The dew points along line C–D are referred to as the upper or retrograde dew points.

The line D–E is sometimes referred to as the normal dew point curve. The dew points along line D–E are referred to as the lower or normal dew points.

Point C is the cricondenbar. It is the highest pressure on the phase envelope. Point D is the cricondentherm. It is the highest temperature on the phase envelope.

**A.6 Limitations of the Phase Diagram**

The accurate determination of a hydrocarbon mixture’s phase behavior depends on the accuracy of the compositional analysis, the equation of state used, the amount of “heavy” (C6+) fractions, and the accuracy of physical properties such as the critical temperature and critical pressure.

These limitations should be considered when using a phase diagram for gas sampling system or method design.
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Figure A.1—Pressure—Volume and Pressure—Temperature Diagrams for a Pure Component
Figure A.2—Pressure—Volume and Pressure—Temperature Diagrams for a Mixture
Figure A.3—Retrograde Condensation
Figure A.4—Examples of Thermodynamic Processes of Natural Gas
Annex B
(Informative)

Fluid Mechanical Considerations in Gas Sampling

B.1 General
It is important to understand the types of flow perturbations that can occur in a pipeline and how these can affect the accuracy of a gas sample. For instance, some piping elements or configurations can create re-circulation zones or eddies in the flow stream. The zone immediately downstream of an orifice plate is a prime example. The gas composition in these eddies may be different from the gas composition of the bulk flow. Other types of flow restrictions or expansions can create localized thermodynamic changes in the gas stream. An example would be the flow from a gas–liquid separator. In that case, the gas will be near its hydrocarbon dew point and a reduction in line temperature will likely cause some condensation to occur, resulting in the flow becoming two phases. In other cases, a pipeline may be operating in a multiphase equilibrium, in which case both gas and liquid are continually present in the pipe. Obtaining an accurate gas sample under these conditions can be quite challenging.

It is not the objective of this document to explain in full detail how flow effects can adversely affect the gas sampling process. There is a large body of information on this subject available in the open literature, to which the reader is referred (see Bibliography). Instead, the following general overview seeks to make the reader aware of potential flow-related problems that may need to be addressed when selecting appropriate gas sampling locations or troubleshooting existing sampling sites.

B.2 Single-phase Flow
The preferred flow regime is single-phase, turbulent flow away from major restrictions to the flowing stream that might produce condensation. Single-phase flow is natural gas flowing at a temperature at or above the hydrocarbon dew point and free of compressor oil, water or other contaminants in the flow stream. In general, it is preferred that the single-phase gas in the pipeline be in the turbulent flow regime because the fluid turbulence creates a well-mixed, representative fluid.

Laminar flow is not normally found in gas pipeline applications because the gas viscosity is relatively low, and the gas velocity is usually high enough to ensure that this flow regime does not occur. However, depending on the design of the gas sampling system, laminar flow can occur in low-flow-rate sampling lines.

Laminar flow is the simplest class of pipe flow, in which streamlines form an orderly flow pattern. A streamline is the trajectory traced out by a moving fluid particle. In laminar flow, viscous forces control the movement of the gas as it moves through the pipe. The gas may be thought of as flowing along in a series of layers or laminates, with smoothly varying velocity from laminate to laminate. There is also no macroscopic mixing of adjacent fluid layers. To illustrate, if a thin film of dye were to be injected into a laminar flow, the dye would appear to be a single line with no dispersion of the dye throughout the flow field (except for slow dispersion due to molecular motion).

Figure B.1 qualitatively illustrates laminar flow at the entrance region of a pipe. In this example, the flow velocity \(U_0\) is uniform at the pipe entrance. The velocity of the gas at the wall of the pipe is always zero and the pipe wall exerts a retarding shear force on the flowing gas. The result is that the gas velocity near the pipe wall is reduced in the axial direction and the effect of the pipe wall is felt farther out in the flow stream as the gas progresses downstream. This phenomenon creates a boundary layer along the pipe wall. At some distance downstream of the pipe entrance, the boundary layer grows to the point that it reaches the pipe centerline. The length required for the boundary layer to reach the pipe centerline is called the entrance length. Beyond the entrance length, the velocity profile does not change with increasing distance and the flow is said to be fully developed. The velocity profile of fully developed laminar pipe flow has a parabolic shape.

Turbulent flow in a pipe is generally characterized by a general swirling nature in the flow field involving indistinct lumps of fluid called eddies or vortices. There is typically a very wide range in the size of the eddies occurring at the
same time or at the same place in the turbulent region. The instantaneous boundary region between the turbulent core flow region and non-turbulent flow region near the pipe wall is sharp. Turbulent flow is also a randomly unsteady process, with effective frequencies ranging over several orders of magnitude. The irregular variations in the motion of the gas stream are not small with respect to either time or space. Furthermore, the turbulence is always three-dimensional, even if the bulk flow is two-dimensional. To illustrate, if a dye filament were to be injected into a turbulent flow, the dye would break up into myriad entangled threads and disperse quickly throughout the entire flow field.

Figure B.2 illustrates the differences in velocity profiles for laminar and turbulent pipe flow. In this case, the pipe Reynolds number (i.e. the non-dimensional ratio of inertial to viscous forces) is $4 \times 10^3$. Both profiles have the same average velocity and, thus, the same flow rate. Notice, however, that the laminar and turbulent velocity profiles do not have the same centerline velocity and that the turbulent profile has a much steeper profile near the wall of the pipe.

Some piping configurations (e.g. headers, single elbows, multiple elbows in series, etc.), flow control elements (e.g. valves, regulators, filters, etc.), or flow metering devices (e.g. orifice flow meters, turbine flow meters, etc.) may distort the gas flow field in a pipe, producing velocity profile asymmetry, swirl, a combination of profile asymmetry and swirl or a phenomenon known as flow separation. Flow separation occurs when the momentum of the fluid in the boundary layer is insufficient and the fluid layers adjacent to the solid surface (e.g. the pipe wall or an obstruction protruding in the pipeline, such as a reduced port valve, flow regulator, or an orifice plate) separate from the surface. Separation results in the formation of a relatively low-pressure region downstream of the separation point or behind the obstruction that produces the flow separation. This low-pressure region is called the wake. Large scale eddies or flow re-circulation zones often develop in wake regions that form in a pipe flow.

These flow distortions may also produce thermodynamic changes (e.g. pressure or temperature changes) that result in non-equilibrium conditions. For instance, thermodynamic effects may cause phase changes in the gas mixture. Care should be taken to select gas sampling locations that are free of flow distortion, since these distortions may cause compositional changes in the gas mixture.

### B.3 Multi-phase Flow

Sampling of multi-phase flow is outside the scope of this standard. Sampling of multi-phase (gas and liquid) mixtures is not recommended and should be avoided if at all possible. In the multi-phase flow, the ideal system would mix the gas and liquid flows uniformly and collect a sample of the true mixture flowing in the line by using a properly designed sample probe and an isokinetic sampling system. Current technology of natural gas sampling is not sufficiently advanced to accomplish this with reasonable accuracy. When sampling a multiphase liquid–gas flow, the recommended procedure is to eliminate the liquid from the sample. The liquid product that flows through the line should be determined by another method. The liquid fraction of the multi-phase flow may contain water and hydrocarbons. The hydrocarbons can contribute significantly to the energy content (measured in British thermal units [Btu]) of the gas and their presence in the gas line should not be overlooked.

![Diagram](image-url)

**NOTE**- Figure excerpted from *Introduction to Fluid Mechanics*, Fox and McDonald.

**Figure B.1—Laminar Flow in the Entrance Region of the Pipe**

64
NOTE- Figure excerpted from *Introduction to Fluid Mechanics*, Fox and McDonald.

Figure B.2—Comparison of Laminar and Turbulent Velocity Profiles for Flow in a Pipe
Annex C
(Informative)

Lessons Learned during Sampling in Hydrocarbon-saturated and Two-phase Natural Gas Streams

During the late 1980s, industry made a commitment to the BLM to complete a full set of consensus standards covering all the measurement-related practices in common use by the industry for custody transfer measurement. As a result of this commitment, several new standards were developed, and several existing standards were upgraded. Examples include standards covering allocation measurement, electronic gas and liquid measurement, Coriolis meters, natural gas sampling, inferred mass liquid measurement, etc.

As a part of the effort, API MPMS Chapter 14.1 was expanded during the early 1990s to include composite sampling of natural gas. During the drafting of the upgraded standard, the Working Group realized that many of the consensus recommendations contained in the standard for both spot and composite sampling were not well supported by sufficient documented and qualified data. Following the balloting and approval of the standard in 1992, a research project was eventually defined and implemented to produce enough high-quality data to make future recommendations technically defensible.

As originally scoped, the research project was to consist of two basic test phases. The Laboratory Phase, conducted at the Southwest Research Institute, was intended to evaluate spot sampling methods under carefully controlled conditions. The Field Phase was intended to evaluate the performance of the most promising sampling methods identified during the Laboratory Phase at actual operating locations. The Field Phase was to be conducted twice, once during summer conditions, then again under winter conditions. The Field Phase was to contain evaluations for composite sampling as well.

Once the project had begun, Colorado Engineering Experiment Station, Inc. (CEESI) commissioned a wet-gas flow facility that offered the Working Group the option of performing the Field Phase under more controlled conditions, but still at operating conditions typical of many field locations. The facility offered the option of performing tests with the flowing stream at, above or below the hydrocarbon dew point of the stream.

The CEESI wet-gas loop provided the ability to blend different compositions of gas in order to evaluate both the repeatability and accuracy of various sampling methods. The gas blends were generally prepared using pipeline gas, then weighing-in additional heavy components as required.

Since the project intended to evaluate methods in the wet-gas loop that were often below the hydrocarbon dew point, many of the data sets were collected under severe operating conditions where it was fully expected that achieving good repeatable analytical results was not likely. The Working Group intentionally designed the tests to penetrate the phase boundary and enter the two-phase region, resulting in entrained and free liquids sometimes being present in the flow loop.

The final scope of the project does not include recommendations for sampling in the two-phase region. The test plan defined by the Working Group for CEESI was performed under such severe conditions in the hope that recommendations could be made concerning the best methods to be used at or near the phase boundary. The data indicated that some methods might be capable of allowing sampling below the hydrocarbon dew point, but with higher uncertainties. The data also clearly demonstrated that under some severe operating conditions, when free liquids are present, none of the current methods are capable of obtaining a representative sample.

Other areas investigated during the course of the research included probe location, spot and composite sampling system design, heating and insulation requirements, laboratory inspection procedures and performance criteria, GPA separators, and visual observation of free liquids (when operating the loop below the hydrocarbon dew point).

The work at CEESI showed that probe location is not critical if flow is single phase (above the hydrocarbon dew point). For flow conditions at or near the hydrocarbon dew point, the probe location should not be placed within or immediately following flow disturbances.
The uncertainty of a portion of the field phase data is high, especially when the gas stream was below the hydrocarbon dew point. However, some data during these periods did reflect a general trend. For example, the constant pressure displacement method tended to be biased slightly low, the fill and empty method appeared to work fairly well as much as 10 °F (6.13 °C) below the hydrocarbon dew point and the helium pop method had a slight negative bias compared to the reference.

In spite of the best intentions of the Working Group and CEESI, there were difficulties in collecting and interpreting the data. Examples include the following:

— more than one uncontrolled variable in some of the test procedures;
— changes in the reference sample point location during the investigations into sample point location and sample method tests;
— calibration problems with the on-line gas chromatograph (peak gates not set properly during the initial test series);
— unstable chromatographic results during consecutive analyses (analyses that failed to reach equilibrium and/or analyses that exhibited poor repeatability);
— large variations in the analyzed nitrogen concentration during various series of analyses;
— sample size variations during analysis due to atmospheric pressure variations and wind effects during the initial test series;
— portions of a single test series being run with the flowing stream at times above and at times below the hydrocarbon dew point temperature;
— lack of knowledge of exactly how the operating conditions related to the phase boundary at different locations within the flow loop during some tests.

Conclusions are as follows:

— When the simulated field conditions were below hydrocarbon dew point, it was difficult to draw clear conclusions from the data. The helium pop and fill and empty methods showed promise under these adverse conditions.
— Trends reflected in the laboratory results were also generally reflected in the simulated field studies, even though the absolute values exhibited a higher uncertainty due to the differences between the reference point and the spot sample method caused by operating below the hydrocarbon dew point in the sample loop.
Annex D
(Informative)

Hydrogen Sulfide Warning

Inhalation of hydrogen sulfide (H₂S) at certain concentrations can lead to injury or death. H₂S is an extremely toxic, flammable gas that may be encountered in the production and processing of gas well gas, high-sulfur-content crude oil, crude oil fractions, associated gas and associated waters. Since H₂S is heavier than air, it can collect in low places in still air. It is colorless and has a foul, rotten egg odor. In low concentrations it is detectable by its characteristic odor. Smell cannot be relied upon to forewarn of dangerous concentrations however, because exposure to high concentrations of the gas (greater than 100 parts per million) rapidly paralyzes the sense of smell. A longer exposure to lower concentrations has a similar desensitizing effect on the sense of smell. **Utilize H₂S monitoring systems to accurately determine H₂S levels.**

Excessive exposure to hydrogen sulfide (H₂S) causes death by poisoning the respiratory system at the cellular level. There is some indication that the presence of alcohol in the blood aggravates the effects of H₂S in acute poisoning cases. At low concentrations (10 to 50 parts per million), H₂S is irritating to the eyes and respiratory tract. Closely repeated, short-term exposures at low concentrations may lead to irritation of the eyes, nose, and throat. Symptoms from repeated exposures to low concentrations usually disappear after not being exposed for an appropriate period of time. Repeated exposures to low concentrations that do not produce effects initially can eventually lead to irritation if the exposures are frequent.

The sense of smell may be rendered ineffective by hydrogen sulfide (H₂S), which can result in an individual failing to recognize the presence of dangerously high concentrations.
Annex E
(Informative)

API/GPA Laboratory Inspection Checklist

The checklist is a tool for evaluating the performance and systems of analytical laboratories.

A critical part of evaluating a laboratory’s performance involves using calibration gas standards to calibrate or verify the calibration of analytical systems. This Annex offers recommendations for using calibration gas standards. Sections 16.2 and 16.3 cover preparing those standards for service.

### API/GPA Laboratory Inspection Checklist

<table>
<thead>
<tr>
<th>Lab Number:</th>
<th>Date:</th>
<th>Survey Team Members:</th>
</tr>
</thead>
</table>

**Sample Handling and Conditioning**

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<tr>
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</tr>
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<tbody>
<tr>
<td>Are sample containers heated?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If sample containers are heated, to what temperature?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the sample container temperature monitored?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the sample container cleaned and purged before use?</td>
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<td></td>
</tr>
<tr>
<td>Is the sample heated for at least two hours?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is time monitored for sample container heating?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What is the length of time used for heating sample containers? (# hours)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Are samples taken immediately from heater to analyzer if manually transferred?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What method is used to insulate heated sample containers during analysis?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Insulated Blanket</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Heated Cabinet</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Other (Specify in Comments)</td>
<td></td>
<td></td>
</tr>
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**Physical Facility**

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Is the analyzer room heated?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the analyzer room air-conditioned?</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Filters, Connections, and Hardware**

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Are filters used between sample and analyzer?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Size:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replacement Interval:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What are the size, length, and material(s) of sample line and fittings?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Are connections, lines, and hardware between sample container and analyzer</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Are connections, lines, and hardware between sample container and analyzer</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample loop size is: 0.25 cc</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.50 cc</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.00 cc</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Other (specify size)</td>
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### Injection System

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<tr>
<th>Question</th>
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<th>NO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Is the sample system a vacuum injection system?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the sample system a purge injection system?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If it is a purge injection system, is there back pressure?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Can the purge rate be read or measured?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What is the purge rate?</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Analyzer

<table>
<thead>
<tr>
<th>Question</th>
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</tr>
</thead>
<tbody>
<tr>
<td>What is the analyzer brand?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What is the analyzer model?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What is the analyzer’s serial number?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is this an isothermal run?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If “YES,” record temperature in °F (°C)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If “NO,” secure a copy of the temperature program.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Are the columns configured per GPA 2261?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If “NO,” list the configuration</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Integration method is:**

- Peak Height:
- Area:
- Data logging:
  - Manual:
  - Electronic:

**Highest carbon number component analyzed is:**

- C6
- C6+
- C7
- C7+
- Other (Specify)

**Calibration schedule is:**

- Daily
- Weekly
- Monthly
- Other (Specify)

**Analysis frequency is:**

- Daily
- Weekly
- Monthly
- Other (Specify)
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### Carrier Gas
<table>
<thead>
<tr>
<th>Question</th>
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<th>NO</th>
</tr>
</thead>
<tbody>
<tr>
<td>What is used as the carrier gas?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>What is the purity of the carrier gas?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the carrier gas pressure monitored?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the carrier gas flow rate monitored?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If yes, carrier gas flow rate in cc/minute:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is a carrier gas drier used?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If yes, type of drier material used:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replacement interval of carrier gas drier material:</td>
<td></td>
<td></td>
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</tbody>
</table>

### Calibration Standard Gas
<table>
<thead>
<tr>
<th>Question</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Manufacturer of calibration standard</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is calibration standard age less than a year old?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If &quot;NO,&quot; list the date blended</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the calibration standard heated continuously?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If no, list the length of time heated before use:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>To what temperature is the calibration standard heated?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is an insulation blanket or heated cabinet used for the calibration standard?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Can the container pressure of the calibration standard be monitored?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If yes, record the pressure in psig (kPa) before and after each test.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Does the lab have calibration standards required for the test program?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Is the hydrocarbon dew point for the calibration standard available?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If yes, hydrocarbon dew point:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Has or could the calibration standard ever been exposed to a temperature below the hydro- carbon dew point?</td>
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### Calculation
<table>
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</thead>
<tbody>
<tr>
<td>Are the component constants used in accordance with the latest GPA 2145?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>If &quot;NO,&quot; what constants are used?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Can the constants be verified?</td>
<td></td>
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</tr>
<tr>
<td>Are the calculations performed in accordance with the latest version of GPA 2172?</td>
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<tr>
<td>Other methods used:</td>
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<td>Values for C6+ or other heavy fraction:</td>
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<td>C6 +</td>
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<td></td>
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<tr>
<td>C7</td>
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<td></td>
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<td>C7 +</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Other (Specify)</td>
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<tr>
<td>Composition of fraction:</td>
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<tr>
<td>C6</td>
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<td>C7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C8 +</td>
<td></td>
<td></td>
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<tr>
<td>Other (Specify)</td>
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</tbody>
</table>
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<table>
<thead>
<tr>
<th>Quality Control Program</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Does a Quality Control Program exist?</td>
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<td></td>
</tr>
<tr>
<td>Can a copy of the Quality Control Program be obtained?</td>
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</table>

**NOTE** Rating by Team

<table>
<thead>
<tr>
<th>Documentation</th>
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<tr>
<td>Secured area counts and response factors?</td>
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<td></td>
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<tr>
<td>Secured chromatograms and results?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Secured copy of analysis report for calibration standards?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Secured relative density?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Secured HV—saturated and unsaturated both real and ideal?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Secured mol % both normalized and un-normalized?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Secured starting and ending pressures for both lab’s calibration standard and audit group’s standards.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**NOTE** Normal heating time for sample containers is 24 hr. (±2 hr.).

**Repeatability and Reproducibility**

See the appropriate GPA standard relative the chromatographic method of interest for the repeatability, reproducibility and precision. e.g. GPA 2261

Repeatability is the expected precision within a laboratory using the same equipment and same analyst. Repeatability is the difference in analyzed values between two sequential runs.

Reproducibility is the expected precision when the same method is used by different laboratories using different equipment and different analysts. Reproducibility is the difference between two analyzed values. Neither value represents the difference between an analyzed value and the certified value listed on a blend.

Performance evaluations commonly use the repeatability and reproducibility of laboratory results compared to a certified blend. The precision statement in a given GPA chromatographic analytical standard is based on the data contained in a round robin relative to a method and the statistical evaluation described in a GPA technical publication (TP) also relative to the same given method. This treatment of data compared laboratory results independent of the certified blend values. Therefore, performance evaluations shall either compare the laboratory results in the same manner by using the reproducibility values described in the relative standard and subsequent example calculations.
Annex F
(Informative)

New Spot or Composite Sample Method Performance Verification Procedure

F.1 General

Testing is limited to a single-phase gas stream at or above its hydrocarbon dew point (HCDP). Multi-phase fluid sampling is not within the scope of this procedure. Laboratory practices described in this document should not be interpreted to be required procedures for normal sampling and analysis.

F.2 Procedure

a) The same chromatograph used to analyze the sample stream is to be used to analyze the spot or composite samples to eliminate the error of using different chromatographs. It is preferable to use chromatographs for testing that are capable of producing an extended analysis (C9+) accurate within the GPA guidelines for repeatability and reproducibility. The use of analyzers limited to a C6+ output may provide test results that indicate that the sampling method is acceptable for the application tested; however, method approval from such results should not be extrapolated to include applications where a more extended analysis is required.

b) Establish that the online or portable chromatograph and sample delivery system complies with this standard. See Annex E for repeatability and reproducibility criteria and inspection checklist. The same calibration method and calibration standard is to be used for all chromatographs used for testing. The GC should be calibrated in the same environment in which it is used to analyze samples, so as to eliminate bias in the analysis.

c) Calibration standards should be prepared in accordance with the requirements as well as maintained in accordance with the requirements in GPA Standard 2198 and verified by a laboratory independent of the blender. Verification of the calibration standard should comply with in Annex E repeatability and reproducibility criteria.

d) Establish that the complete sampling system (chromatograph, containers, tubing, etc.) is clean and free of any contaminants prior to calibrating and testing. Verify that the sample delivery system and chromatograph are clean by analyzing a sample of ultra-high purity (UHP) helium. Verify the cleanliness of the sample containers by charging them with 50 psig (345 kPa gauge) of UHP helium, heating to 125 °F (52 °C) for 2–4 h, and then analyzing the helium. No peaks should be produced during these procedures.

e) If the sample method is tested under controlled laboratory conditions [as in e)(1)], performance verification is recommended on multiple gas blends. If the method is tested on a single flowing gas composition at an established metering location [as in e)(2)], the results may not apply to a broad spectrum of gas compositions.

The protocol is intended to evaluate the performance of new or proposed sampling systems and procedures under both ideal conditions and under those conditions when only the best current methods will succeed. For example, in a case where two gases are used during the evaluation, the lean gas may be used to verify good performance when the gas is far from its hydrocarbon dew point (HCDP) and ambient temperatures (real or induced) are higher than the flowing temperature of the stream. The richer gas, during the same evaluation, would be used to evaluate the performance of the new or proposed sampling system and procedure when the ambient temperature is colder than the flowing temperature of the stream and the measured hydrocarbon dew point of the stream and the flowing stream is at a temperature very near its HCDP.

1) Verify the method on at least two and preferably three gas blends that represent a broad spectrum of gas compositions commonly encountered in gas gathering and transportation operations. For example, a three-gas test might use blends that produce gross heating values of 1000 Btu/scf, 1175 Btu/scf, and 1350 Btu/scf.
2) Select a location for sampling that has a steady flow rate and a stable gas composition. Verification of stability will be established before sampling is conducted. Stability is defined as the repeatability of consecutive analyses as defined in Annex E of this document.

3) To fully confirm the suitability of new or proposed sampling systems and procedures, it is recommended that the flowing stream during one phase of the evaluation program be within 5 °F (2.8 °C) of its’ measured HCDP and that the ambient temperature (actual or induced) be at least 20 °F (11.1 °C) colder than the flowing temperature of that stream.

f) Measure the HCDP using the following steps. See Bibliographic Reference 9 for further guidance.

1) Use a Bureau of Mines–type dew point apparatus with a NIST-traceable temperature-measuring device, preferably with a video attachment.

2) Heat the sample line and the Bureau of Mines–type dew point apparatus to at least 20 °F (11.1 °C) above the temperature of the flowing stream or gas source.

3) The dew point is to be measured before, during, and after the sampling procedure tests.

g) Determine the composition of the flowing stream using the following steps.

1) Verify and document the stability of the composition of the flowing gas stream using a portable or on-line chromatograph.

2) The chromatograph is to be analyzing the stream during sampling procedures.

3) The chromatograph software will be required to archive, and time and date stamp the chromatograms, composition, and the corresponding heating value. The methods and calibration chromatograms used for each testing procedure shall be saved.

h) Also capture and record sufficient data to demonstrate the stability of the dynamic flowing conditions of the stream during the sampling tests. All instruments that record data with time stamps, such as flow meters, gas chromatographs or data loggers, should be synchronized to avoid errors in data comparisons.

i) Conduct sampling method using the following steps.

1) A minimum of five (5) samples shall be obtained for each method tested.

j) Analysis Requirements

1) Each container shall be heated to 50 °F (27.8 °C) above the HCDP for a period of four hours before analyzing.

2) Each container is to be analyzed a minimum of three times to establish repeatability as defined in Annex E of this document.

k) Data Requirements

1) The data shall be in tabular form and in a format that is easy to read and understand.

2) The data shall be available for public review.

3) The spot sample analysis report shall reference the corresponding on-line or portable gas analysis report.
4) On-line chromatograph data for comparison with composite samples shall be averaged for the same time period as the composite samples.

1) Verification of sampling procedure: Sample heating values shall agree within the greater of the tolerances described below or those defined in Annex E.

1) Review the data to ensure that the accuracy of the spot or composite sample method complies with the repeatability and reproducibility for each component as stated in Annex E.

2) The three successive analyses of each test container should repeat within one (1) Btu per cubic foot (37.3 kJ/m³).

3) Each test container should reproduce within three (3) Btu per cubic foot (111.7 kJ/m³) of the on-line or portable chromatograph.

4) Analyses of each composite sample container should reproduce within (3) Btu per cubic foot (111.7 kJ/m³) of the average of the on-line chromatograph for that sampling period.

Record the following Information. Description of the sample method:

Description of the test used to evaluate the sample method:

Date of test:
Location of the test:
Personnel conducting the tests:
Personnel witnessing the tests:
Pipeline pressure:
Pipeline size:
Piping configuration (upstream and downstream):
Sample probe style and description:
Sketch or photo of the piping scheme for the test site:
Description or photo of the test method equipment and physical installation used in test:
Description of the flowing gas stream, i.e. well head, separator, distribution system, meter run, gathering system, etc.:
Ambient temperature:
Pipeline flowing temperature:
Sampling apparatus temperature for sample method and reference analyzer.

As a minimum, include the temperature at the sample probe and at the exit point of the sample delivery system:
Description of temperature control equipment (i.e. insulation, heater, steam):

Hydrocarbon dew point:

Container style:

Container size:

Sampler style (if applicable) and description:

Timed or proportional to flow:

Sample volume size:

Approximate elevation of the test location:

GC description:

Last calibrated and how, relative to test data:

Provide records for calibrations performed immediately before, during and after the testing, including response factors and repeatability data.

Record test data on a spreadsheet and provide as much additional information as possible, including copies of analyses from laboratories.
Annex G
(Informative)


G.1 Purpose and Scope

This Annex provides guidance for determining the hydrocarbon dew point temperature of a natural gas stream using a manually operated Bureau of Mines–type chilled mirror dew point apparatus. This Annex applies to hydrocarbon dew point measurements of single-phase natural gas streams that are at or above their hydrocarbon dew point temperature. This Annex is not intended for determination of the water vapor dew point temperature of natural gas. Reference ASTM Standard D1142 for details on water vapor dew point determination.

G.2 Normative References


G.3 Dew Point Apparatus

G.3.1 Design Requirements

Any properly constructed chilled mirror dew point apparatus can be used that satisfies the following basic requirements.

a) The apparatus is designed to permit a controlled flow of gas to enter and leave the apparatus while it and the sampling system are at a temperature of at least 30 °F above the flowing temperature of the gas within the pipeline.

b) The apparatus shall provide sufficient control of the refrigerant cooling rate to lower the mirror surface to a temperature at which hydrocarbons will condense and an accurate dew point temperature can be determined.

c) A visual port allows observation of the deposition of hydrocarbon condensate within the apparatus.

d) The apparatus provides a means to measure the temperature of the cold portion of the apparatus on which the hydrocarbon condensate is deposited.

e) The apparatus provides a means to measure the pressure of the gas within the apparatus.

f) The apparatus should be constructed so that the “cold spot”, i.e. the cold portion of the apparatus on which the hydrocarbon condensate is deposited, is exposed only to the gas under test.

G.3.2 Bureau of Mines Manual Chilled Mirror Dew Point Apparatus

The Bureau of Mines–type of chilled mirror dew point apparatus shown in Figure G.1 fulfills the requirements specified in G.3.1. Within the scope of conditions listed in G.1, this apparatus is satisfactory for determining the hydrocarbon dew point temperature of gaseous fuels.
The apparatus consists of a metal test chamber into and out of which the test gas is permitted to flow through control valves D and A. Gas entering the apparatus through valve D fills the test chamber, flows across the mirror, C, and flows out of the test chamber through valve A. Part C is a highly polished stainless steel “target mirror,” cooled by means of a copper cooling rod, F. The mirror, C, is silver-soldered to a nib on the copper thermometer well fitting, I, which is soft-soldered to the cooling rod, F. The thermometer well is integral with the fitting, I. Cooling of rod F is accomplished by vaporizing a refrigerant such as liquid butane, liquid propane, carbon dioxide, or some other liquefied gas in the chiller, G.

The refrigerant is throttled into the chiller through valve H and leaves through outlet J. The chiller body is made of copper and has brass headers on either end. The lower header is connected with the upper header by numerous small holes drilled in the copper body, through which the vaporized refrigerant passes. The chiller is attached to the cooling rod, F, by means of a taper joint. The temperature of the target mirror, C, is indicated by a calibrated glass type of thermometer, K, whose bulb fits snugly into the thermometer well. A calibrated resistance temperature detector (RTD) can be used instead of a glass thermometer, with the sensing end of the RTD placed in the thermometer well to measure the mirror temperature. When an RTD or similar temperature measurement device is used, it shall include

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Conical valve, gas outlet</td>
</tr>
<tr>
<td>B</td>
<td>Pressure gauge</td>
</tr>
<tr>
<td>C</td>
<td>Mirror</td>
</tr>
<tr>
<td>D</td>
<td>Control valve, gas inlet</td>
</tr>
<tr>
<td>E</td>
<td>Window</td>
</tr>
<tr>
<td>F</td>
<td>Copper cooling rod</td>
</tr>
<tr>
<td>G</td>
<td>Chiller</td>
</tr>
<tr>
<td>H</td>
<td>Refrigerant control valve</td>
</tr>
<tr>
<td>I</td>
<td>Well fitting</td>
</tr>
<tr>
<td>J</td>
<td>Refrigerant outlet</td>
</tr>
<tr>
<td>K</td>
<td>Thermometer</td>
</tr>
</tbody>
</table>

Figure G.1—Diagram of a Typical Bureau of Mines Chilled Mirror Dew Point Apparatus
suitable electronics and a temperature readout device for the user. Observation of the deposit of condensate is made through the pressure-resistant transparent window, \( E \).

Note that only the central part of the stainless steel target mirror, \( C \), is thermally bonded to the fitting, \( I \), through which \( C \) is cooled. Since stainless steel is a relatively poor thermal conductor, the central portion of the mirror is thus maintained at a slightly lower temperature than the outer portion, with the result that the dew first appears on the central portion of the mirror and its detection is aided materially by the contrast afforded.

The arrangement for measuring the temperature of the target mirror, \( C \), should be noted. The temperature is read with a thermometer, \( K \), or an RTD inserted in the cooling rod, \( F \), so that the bulb or sensing end of the temperature measurement device is entirely within the thermometer well fitting, \( I \). The stud to which the stainless steel mirror is silver-soldered is a part of the base of the thermometer well. As there is no metallic contact between the thermometer well and the cooling tube other than through its base, the thermometer or RTD indicates the temperature of the mirror. This design avoids the measured temperature being influenced by the temperature gradient along the cooling tube.

G.4 General Considerations for Accurate Dew Point Temperature Measurement

Before any measurements of hydrocarbon dew point begin, a number of considerations shall be addressed to ensure an accurate dew point temperature measurement. The process begins with selection of a test location that will produce a representative sample of the gas stream. Any location used to draw samples for dew point temperature measurements should meet the requirements addressed in this standard. When dew point temperatures are being measured to prepare for the collection of natural gas samples, the dew point measurements and the gas samples should be taken at the same location.

The use of properly designed sample probes to take representative samples of the flowing gas stream is essential in the practice of sampling. Specific to the dew point apparatus application, regulating or filter type probes shall not be used as they can interfere with measurements. Materials used in the construction of sample transfer lines should be selected to avoid adsorption of gas components and potential sample distortion.

Pressure and temperature changes between the pipeline and the dew point apparatus that can cause condensation shall be avoided. Within the dew point apparatus, measurements should be made at the pressure of the flowing gas stream. The temperature of the sample line from the flowing pipeline to the dew point apparatus should be maintained at or above the temperature of the pipeline flow.

When ambient conditions could cause cooling of the sample stream between the flowing pipeline and the dew point apparatus, proper use of insulation, heat trace or other heating equipment should be employed. Devices that cool the gas stream through Joule–Thomson (J–T) cooling, or that throttle the gas flow between the pipeline and the dew point apparatus, should be avoided.

Determining the flowing gas temperature and pressure at the dew point sampling location is critical to accurate measurement of the dew point temperature. When the dew point testing equipment indicates that the dew point temperature is at or above the flowing line temperature, the presence of liquids in the pipeline should be assumed. Pressure and temperature measuring instruments used with the chilled mirror dew point apparatus to determine hydrocarbon dew point shall be verified and calibrated at appropriate intervals not to exceed two years or as recommended by the instrument manufacturer; whichever period is shorter.

Consideration shall be given to applying the correct flow rate through the dew point apparatus. The temperature at which a visible droplet first forms on the mirror depends not only on the mirror temperature and the pressure at which the measurement is taken, but also on the rate of temperature change of the gas. At slower flow rates across the chilled mirror, the gas will cool more quickly, and is more likely to reach the mirror temperature. The rate of temperature decrease of the sample gas inside the apparatus will impact the rate of liquid condensation and the potential accuracy of the dew point temperature measurement. The potential for J–T cooling due to pressure change when sample gas passes through the inlet valve, control valve \( D \), should also be addressed. Too large of a pressure decrease, caused by high flow rates through the valve, can cause liquids to condense from the sample gas before reaching the mirror surface, biasing the measurement (see G.7.3).
G.5 Safety Considerations

Before starting a dew point test, inspect all parts of the apparatus for damage and wear. The inspection should include, but not be limited to, the chilled mirror dew point apparatus, sample delivery lines, refrigerant lines, and the valve at the sample point. Worn O-rings or loose fittings can pose safety hazards and shall be replaced or tightened.

Natural gas streams can be at high pressure. Refrigerant, such as re-gasified liquid nitrogen or carbon dioxide, or propane can be at extreme temperatures and/or stored at high pressure. Be aware of and follow all industrial safety practices concerning pressurized gases.

Natural gas streams can contain components that are toxic or hazardous (H₂S, benzene, toluene, etc.). Sample and refrigerant gases can displace the breathable air supply in small spaces. Consideration shall be given to proper ventilation when working in a confined space.

Natural gas streams can be at extreme temperatures, either hot or cold. Refrigerant gases can be stored at extremely cold temperatures. Contact with skin can cause severe burns, and proper precautions should be taken to avoid contact with natural gas streams or refrigerants.

Natural gas streams can be flammable or explosive. All electrical or electronic devices used in dew point measurements shall be rated for the working environment. Be aware of possible ignition sources in the area (static electricity, cell phones, open flames, non-Class I/Div. I electronics, flare stacks, etc.). A chilled mirror dew point apparatus shall not be used in oxygen service applications.

When using removable sample probes to draw samples from a flowing pipeline, always follow the manufacturer's directions.

Caution- Improper use of these units can cause serious injury or death.

G.6 Procedure

G.6.1 General Considerations

Hydrocarbon dew point temperatures shall be measured at actual line pressure whenever possible. The pressure and temperature measurement devices used with the chilled mirror dew point apparatus shall demonstrate accuracy appropriate to the requirements of the test.

Before tests begin, both the window and the mirror, items E and C in Figure G.1, should be cleaned with a cotton ball, cotton swab, or a clean, lint-free soft cloth. Deposits on the mirror can act as nucleation sites, which can bias the hydrocarbon dew point temperature measurement. Both the window and mirror can be cleaned with isopropyl alcohol or with soap and water followed by isopropyl alcohol. Care should be taken to prevent scratching these surfaces. A window which is scratched, etched, or the integrity of which is otherwise compromised should be replaced immediately as the window can be prone to catastrophic failure under pressure or visibility can be obscured. Scratches on the mirror can also act as nucleation sites for condensation, which can bias the measured hydrocarbon dew point temperature.

Some dew point apparatuses include internal lights to illuminate the mirror, while others illuminate the mirror using ambient light. Using an ambient light apparatus to observe the dew points can present problems due to conditions at the test location (time of day, bright or cloudy conditions). Using an ambient light apparatus can also have some advantages. For instance, the operator can change the lighting conditions on the mirror or change the angle of observation by moving his or her head. On some apparatuses, the mirror can also be moved to improve the observation of events inside the chamber. With either design, the operator is still looking for the same events to occur on the mirror as the temperature is slowly lowered. No matter what type of optical device is used, practice and experience will lead to accurate temperature measurement and operator confidence.
Hydrocarbon dew point temperatures can only be accurately detected on a brightly lit mirror. This is the opposite of the case for water vapor dew point temperatures, which are best detected on a “dark” or dimly lit mirror. When the dew point apparatus has been adjusted for water vapor measurement, so that the mirror is “dark,” the light source or mirror angle shall be changed to brighten the mirror as much as possible.

As the hydrocarbon dew point temperature is reached, small liquid nucleations will form on the brightly lit mirror. The temperature at the moment these nucleations are observed is defined as the hydrocarbon dew point temperature. The operator shall record the temperature when droplets are first observed on the mirror. When the mirror is cooled past the dew point, the hydrocarbon nucleations will become larger and eventually pool together, forming large droplets on the mirror surface. When cooling continues, these droplets will start to run down the surface of the mirror. Eventually, the mirror will be coated with a bright sheen of hydrocarbon liquid, and a large pool will form at the bottom of the mirror. It is recommended that the mirror be cooled past the recorded hydrocarbon dew point during tests, to confirm that the observed dew point is actually a hydrocarbon dew point, and not water or some other liquid condensation.

G.6.2 Detailed Procedure for Operation of the Chilled Mirror Dew Point Apparatus

Introduce the gas sample through inlet control valve D (see Figure G.1), fully opening this valve so that full line pressure is introduced into the apparatus. Per manufacturer recommendation, control the flow through the outlet valve, A, so that there is not a measurable pressure drop in the dew point apparatus, approximately a rate of 5 standard cubic feet per hour (SCFH) for pipeline pressures less than 1000 psig or 2 SCFH for pipeline pressures above 1000 psig to reduce the J–T effect. Allow the gas sample to flow through the apparatus until the system reaches thermal equilibrium as indicated by a stable temperature read at the thermometer, K, for several minutes.

With liquefied refrigerant piped to the chiller throttle valve, H, slightly open the throttle valve momentarily, allowing the refrigerant to vaporize in the chiller and produce suitable lowering in temperature of the chiller tube, F, and target mirror, C, as indicated by the temperature-measuring device, K. Slowly lower the temperature of the target mirror by opening and closing the throttle valve so that the cooling rate does not exceed 1 °F/min (0.5 °C/min). Continue to lower the temperature of the mirror until condensation is observed and the dew point temperature has been determined.

The rate of cooling can be as rapid as desired in making a preliminary test. After estimating the hydrocarbon dew point temperature from the preliminary test or from some other knowledge, subsequent tests should be performed at a cooling rate of 1 °F/min (0.5 °C/min) or less. When the temperature is lowered too rapidly, an accurate measurement is not possible (see G.7.3.2). Tests should be repeated to confirm the accuracy of the initial measurement. To repeat the test, warm the device until the hydrocarbon condensation disappears, then repeat the process of lowering the mirror temperature.

G.6.3 Identification of Hydrocarbon Dew Point Temperatures and Visual Examples

Chilled mirror dew point apparatuses can be used to determine both water vapor dew points and hydrocarbon dew points. The two types of dew points can be distinguished from one another by the unique location and size of the liquid droplets that form on the mirror surface. These differ because of differences in surface tension between liquid water and liquid hydrocarbons. In either case, the dew point temperature is defined as the temperature at the instant condensation is sighted on the mirror.

Because dew points are visually identified, operator experience is beneficial in determining dew point temperatures repeatedly and efficiently, and to distinguish hydrocarbon condensation from other types of condensation on the chilled mirror. The photographs in Table G.1 and Table G.2 are examples of the different formations that can be seen on chilled mirrors and can be used as a guide. Often, an iridescent ring can be seen on the mirror before hydrocarbons condense. This is a precursor to the hydrocarbon dew point but does not mark the dew point temperature itself. Also, it is possible to observe a combination of dew points simultaneously. Experience will allow operators to identify different dew point temperatures of the natural gas stream reliably.
G.7 Measurement Uncertainties

G.7.1 General

Test results using the Bureau of Mines chilled mirror dew point apparatus are subject to the visual interpretation of the condensation on the mirror surface. Dew point temperature measurements depend on the operator's skill at controlling the refrigerant flow rate and on the operator's ability to identify and interpret the condensation seen on the mirror. This section discusses uncertainties in dew point temperatures measured with the Bureau of Mines manual apparatus, some of which result from differences between operator observations and techniques.

Table G.1—Examples of Hydrocarbon Dew Formations on a Chilled Mirror Surface

<table>
<thead>
<tr>
<th>Description</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Clean mirror:</strong> appears smooth and clean, with no nucleation sites. The mirror shown in this photo is darkly lit for water vapor detection. For hydrocarbon detection, the mirror should be bright, shiny, and clean.</td>
<td></td>
</tr>
<tr>
<td><strong>Iridescent ring:</strong> a precursor to the hydrocarbon dew point, but not the dew point itself. Rainbow-like colors form in the center of the mirror and quickly expand to cover the mirror. Usually, but not always, this forms before the hydrocarbon dew point temperature is reached.</td>
<td></td>
</tr>
<tr>
<td><strong>Hydrocarbon dew point:</strong> at the exact hydrocarbon dew point temperature, small droplets or nucleations form on the mirror within the iridescent ring.</td>
<td></td>
</tr>
<tr>
<td><strong>Intermediate hydrocarbon condensation:</strong> as the temperature falls, more droplets form.</td>
<td></td>
</tr>
</tbody>
</table>
**Advanced hydrocarbon condensation:** droplets collect into large drops that can run down the side or edges of the mirror.

**Hydrocarbon ice:** a white frost that forms at temperatures well below the hydrocarbon dew point.

---

**Table G.2—Examples of Other Condensations on a Chilled Mirror Surface**

<table>
<thead>
<tr>
<th>Condensation Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Water vapor dew point</strong></td>
<td>at the exact dew point temperature, a small gray spot appears near the center of the mirror.</td>
</tr>
<tr>
<td><strong>Advanced water vapor condensation</strong></td>
<td>as the temperature falls, water vapor forms a distinct, opaque gray spot with sharp edges, directly in the center of the mirror.</td>
</tr>
<tr>
<td><strong>Alcohol dew point</strong></td>
<td>a white spot forms in the center of the mirror, but with indistinct edges, unlike the sharp edges of the gray, opaque water dew point. The spot might have a blue tint around the edges.</td>
</tr>
</tbody>
</table>
Advanced alcohol condensation: the entire mirror becomes very white until droplets form. The drops become larger, but continue to cling to the mirror without freezing.

Glycol contamination: Glycol condensation will form a light haze covering the entire mirror, but will not evaporate when the mirror is reheated. When glycol dew forms on the mirror, disassemble the dew point apparatus and clean the mirror and tubing as discussed in G.6.1.

G.7.2 Precision Uncertainty

The temperature of the chilled mirror is typically measured with a thermometer or with an electronic instrument such as an RTD. Such instruments will have a precision measurement uncertainty associated with them. These can be due to random influences on the measurement device and can also depend on its measurement resolution.

When the measurement device has been calibrated, the precision measurement uncertainty and its confidence level shall appear on the calibration certificate for the device. Precision measurement uncertainties should also appear in the instructions for the chilled mirror dew point apparatus itself.

G.7.3 Potentials for Bias

G.7.3.1 Bias Due to the Limit of Visibility of Condensation. One study (Cowper, 2002)\textsuperscript{[G.3]} estimated a systematic bias error in chilled mirror measurements of dew point temperatures. Very thin films of condensation on the chilled mirror will not be visible to an operator when the thickness of the liquid is comparable to a single wavelength of visible light. The study estimated the minimum film thickness that would be visible on a typical chilled mirror. The study then determined the amount of cooling, below the “analytical” dew point temperature at which the first condensation forms, at which various gases would form a visible amount of liquid.

For a typical chilled mirror dew point apparatus, a dew point temperature measurement was estimated to underestimate the “analytical” dew point temperature by 0.4 °F to 2.7 °F (0.2 °C to 1.5 °C). This negative measurement bias depended mainly upon the properties of the gas being analyzed.

G.7.3.2 Bias Due to Rapid Chilling of the Mirror. A bias can be introduced to the test results when the 1 °F/min cooling rate of the apparatus is exceeded because the temperature recording can lag behind the actual mirror surface temperature. This can be expected to cause a positive bias in measured dew points. The faster the cooling rate, the larger the time lag, and the larger this positive bias is expected to be. No estimates of this bias are available but it can be expected to vary with the thermal response of the temperature measurement device used.

G.7.4 Combined Uncertainties

G.7.4.1 Measurement Reproducibility. A round-robin study (Warner et al., 2001)\textsuperscript{[G.4]} estimated the overall reproducibility of hydrocarbon dew point temperatures measured with the chilled mirror dew point apparatus. As part of
the study, several operators, with a wide variety of experience, independently measured the dew point temperatures of lean natural gases using different chilled mirror apparatuses. Statistics were used to find the reproducibility of dew points among the different operators.

The reproducibility of hydrocarbon dew point temperatures from all the operators was found to be ±2.3 °F (±1.3 °C) at the one-sigma confidence level. This value depended mainly upon differences between the operators performing the tests and included contributors such as different chilling rates and visual interpretation. This value did not include the bias due to the limit of visibility discussed in G.7.3.1. Differences in the chilled mirror dew point apparatuses themselves were also found to have a negligible effect on the reproducibility figure.

G.7.4.2 Comparison to Calculated Estimates. In the same round-robin study, the chilled mirror dew point measurements were compared to “known” dew point temperatures of the gases. The “known” dew points were estimated from equations of state and round-robin GC analyses of the gases at different laboratories. These comparisons were used to estimate the degree of accuracy of the measurements.

The agreement between calculated estimates and the dew point determinations from all the operators was found to be ±2.4 °F (±1.3 °C) at the one-sigma confidence level. The agreement with calculated estimates is influenced by those factors mentioned in G.7.2 along with any other uncertainties including those associated with calculating a dew point utilizing an analysis and equation of state estimation.

For a given cooling rate, the bias due to sub-cooling can be greater for measurements at pressures below the pressure of the cricondentherm and/or for gases with components having molecular weights greater than the molecular weight of normal decane (Starling, 2007)\textsuperscript{[G.5]}. 

G.9 Bibliographical References for Annex G


Annex H
(Informative)

Gas Sampling Checklists

### H.1 GAS SAMPLING FIELD CHECKLIST

Users of this Checklist should not rely exclusively on the information contained in this document. Sound business, scientific, engineering, and safety judgment should be used in employing the information contained herein.

<table>
<thead>
<tr>
<th>GENERAL CONSIDERATIONS</th>
<th>YES</th>
<th>NO</th>
<th>RECORD</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Are Operating Conditions Representative Of Normal Flowing Operations?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 Flowing Temperature</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>3 Flowing Pressure</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>4 Flow Rate</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>5 Ambient Temperature</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
</tbody>
</table>

(Representative of typical sources and operating conditions)

Note: For new installations, the system may need to flow a few days to ensure samples are representative.

<table>
<thead>
<tr>
<th>GENERAL SAMPLING INFORMATION</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>6 Sampling Frequency</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7 Site/Facility (Include Sample Point Location Identifier/Descriptors)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 Date?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9 Time?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 Sample Temperature?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11 Sample Pressure?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12 Collection Method?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13 H2S Present? Amount? (When H2S or other sulfur compounds are found or known to be present refer to H.2 Line #3.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14 Special Considerations?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>15 Person Collecting The Sample Identified?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>16 Samples Properly Labeled And Marked?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>17 Samples Transported Safely?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>18 Well Marked For Flammability And Sampled Fluid?</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>19 Valves And Valve Threads Protected?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>20 Proper Rack Or Carrying Case?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>21 Threads Taped To Prevent Leakage?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>22 Conduct Leak Test Of Sampling System?</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

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### H.1 Gas Sampling Field Checklist (Continued)

#### SPOT SAMPLING

A Note to the User: All methods referenced in API 14.1 are valid and acceptable methods for sample collection and may not necessarily be best covered by this checklist; in those cases the user is directed back to Section 12.1 of the main document.

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
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</thead>
<tbody>
<tr>
<td>23</td>
<td>For Spot Sampling, Particularly At Or Near The Hydrocarbon Dewpoint, Is The Fill And Empty Method Being Utilized? (See API 14.1, Section 11.8.)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>24</td>
<td>For The Fill And Empty Method, Does The Apparatus Meet The Recommended Design? (See API 14.1, Figure 9 In Section 11.8.)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>For The Fill And Empty Method, How Many Times Was The Cylinder Purged? Did The Number Of Purges Meet API 14.1? (See API 14.1, Table 2 In Section 11.8.)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>26</td>
<td>For Spot Sampling, Particularly At Or Near The Hydrocarbon Dewpoint, Is Helium Pop Method Being Utilized? (See API 14.1, Section 11.4.)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>27</td>
<td>For Helium Pop Method, Does The Apparatus Meet The Recommended Design? (See API 14.1, Section 11.4.)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>28</td>
<td>Do Sample Cylinder And Probe Valves Have 1/8 Inch Ports Or Larger?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>Has Sample Cylinder Been Purged And Cleaned Prior To Being Used?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>For The Fill And Empty Method, Is The Extension Tube At Least 36 Inches In Length (For Easy Handling May Be Coiled With No Metal On Metal Contact Between Coils. The Pigtail Outlet Or Orifice Must Face Downward)?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>31</td>
<td>Is An Extension Tube Utilized With The Fill And Empty Method Equipped With An Outlet Valve With A Port Ranging From 1/16 To 1/8 Inch Diameter?</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

#### COMPOSITE (See API 14.1, Section 12.1) AND CONTINUOUS SAMPLING SYSTEMS (See API 14.1, Section 12.2)

<table>
<thead>
<tr>
<th></th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>32</td>
<td>Has Sample Cylinder Been Purged Prior To Being Used?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>33</td>
<td>When Composite Sample Cylinders Are Changed Out, Is The Sample Loop Purged To Remove Air And Non-Representative Gas From The System Prior To Restarting The System?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>34</td>
<td>When Installing A New Sample Cylinder In A Composite Sampling System, Was The Sample Cylinder Properly Conditioned / Purged (When Using A Single Cavity Cylinder Was The Cylinder Purged In A Vertical Position)?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>Are Composite And Continuous Sampling System Purge Cycles Adequate To Ensure Fresh Sample Is Available To Composite Or Continuous Sampling Systems?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>36</td>
<td>For Composite Sampling Systems, Has The Stroke Volume And Stroke Frequency Been Carefully Considered To Ensure Sample Cylinders Are Not Over-Filled Before The Collection Cycle Ends?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>37</td>
<td>For Composite Sampling, Is The Sample Taken Proportional-To-Flow?</td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<td>---</td>
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<td></td>
</tr>
<tr>
<td>38</td>
<td>Is A Sample Probe Provided?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>39</td>
<td>Is Sample Probe Located Near The Associated Metering System?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>Is Sample Probe Within 45 Degrees Of Vertical In A Horizontal Line?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>41</td>
<td>Is The Sample Probe At Least 5 Pipe Diameters Downstream Of A Major Disturbance (Such As Orifice Plates, Elbows, Tees, Reduced-Port Valves, Flow Conditioners, Fillers, Strainers, Etc.)? See API 14.1, Section 6.4.2.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>42</td>
<td>Is The Sample Probe At Least 5 Diameters Downstream Of A Minor Disturbance (The Maximum Diameter Of The Disturbance)? See API 14.1, Section 6.4.2.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>43</td>
<td>Is The Sample Probe Beyond The Required Upstream And Downstream Straight Pipe Lengths Required For The Flow Measurement Device In Use?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>44</td>
<td>Does The Sample Probe Extend Into The Center 1/3 Of The Line?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>45</td>
<td>Is The Sample Probe Designed To Adequately Withstand The Maximum Anticipated Flow Rates In The Flowline? See API 14.1, Table 1 In Section 6.4.1.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>46</td>
<td>Is Meter Run Located In An Enclosure Or Building?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>47</td>
<td>Is The Sample Probe Also Inside The Enclosure Or Building?</td>
<td></td>
<td></td>
</tr>
<tr>
<td>48</td>
<td>If Sample Probe Is Not In Enclosure Or Building, Is It In An Insulated Section Of Line Adjacent To The Run?</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

For runs inside enclosures, the sample probe should be at the same operating conditions as the meter. In accordance with API 14.1 Section 6.3.1 both straight and angle cut probes are acceptable; straight cut is preferred but not required for proper sample collection. If angle cut, the direction of the angle cut is not significant.

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>49</td>
<td>For Spot Sampling, Is The Sample Cylinder Close-Coupled With The Sample Probe?</td>
<td></td>
</tr>
<tr>
<td>50</td>
<td>For Sampling To A Portable GC, Is A Speed Loop Designed To Produce A Fresh Sample To The GC? See API 14.1, Section 12.2.</td>
<td></td>
</tr>
<tr>
<td>51</td>
<td>Are Sample System Components Stainless Steel?</td>
<td></td>
</tr>
<tr>
<td>52</td>
<td>Does Tubing Used In The Installation Meet API Chapter 14.1 Recommendations? See API 14.1, Section 5.3.2.3.</td>
<td></td>
</tr>
</tbody>
</table>

### HEAT TRACING AND INSULATION

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>53</td>
<td>Is Heating Provided To Prevent Condensation In The Sample System? (Especially During Pressure Reduction.)</td>
<td></td>
</tr>
<tr>
<td>54</td>
<td>Has Multi-Stage Regulation For High Pressure Flowing Streams Been Provided (For Safety And For Effective Heat Tracing)?</td>
<td></td>
</tr>
<tr>
<td>55</td>
<td>Where Utilized, Does Heat Tracing And Insulation (Including Any Conditioned Enclosures) Extend From The Sample Probe To The Inlet Valve Of The Composite Sample Cylinder Or To The Injection Valve Of The Onstream Analyzer?</td>
<td></td>
</tr>
</tbody>
</table>
H.2 GAS SAMPLING LAB CHECKLIST
(Refer to Appendix E for Complete Laboratory Checklist)

Users of this Checklist should not rely exclusively on the information contained in this document. Sound business, scientific, engineering, and safety judgment should be used in employing the information contained herein.

<table>
<thead>
<tr>
<th>GENERAL LAB CHECKLIST</th>
<th>YES</th>
<th>NO</th>
<th>N/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Is The Stream Single Phase Gas? (If Unknown See API 14.1, Section 4.2.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 For Sampling Under Low Pressure Or Vacuum Conditions, Does The Sample Cylinder Have Sufficient Volume And Pressure To Be Analyzed? Note: Non-lubricated sample pumps or inert gas repressurization may be required.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3 For Sampling Of Gas Streams Containing Very Reactive Components (ex. H2S) Have The Sample Cylinders Been Internally Lined Or Coated To Ensure Sample Integrity? (See API 14.1, Section 9.1.) Note: Very reactive components, such as hydrogen sulfide (H2S), should be analyzed on-site when practical.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 Were Sample Cylinders Adequately Cleaned And Conditioned Prior To Use?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 Is An Optional Inert Gas Blanket Added To The Cylinder After Cleaning? (See API 14.1, Section 5.5.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6 Are &quot;Clean&quot; Cylinders Randomly Subjected To Periodic Chromatograph Runs To Validate There Are No Hydrocarbon Peaks Upon Sample Analysis?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>7 Are Sample Cylinders Adequately Heated For A Period Of At Least Two Hours Prior To Analysis? (See API 14.1, Section 5.6.4.) Note: Sample cylinders should be heated to at least 30 °F above the higher of the flowing temperature marked at time of sample collection or the hydrocarbon dew point temperature.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 Were Sample Cylinders Heated For At Least 2 Hours Prior To Analysis Or Withdrawals?</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>9 Were Calibration Standard Cylinders Heated For At Least 4 Hours Prior To Any Withdrawals?</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

[Formatting note: Tables in Annex H will be updated during editing.]
Annex I
(normative)

Cleaning

General Cleaning Requirements

All methods in this document require that the sample containers and associated sampling equipment be clean and free of leaks.

When gasoline-range hydrocarbons are present in a container prior to sample procurement, the sample can become enriched, yielding an elevated heating value.

Heavier contaminants such as oil or glycol can absorb heavy components of the sample, reducing the heating value of the sample.

Not all cleaning methods remove both of these ranges of contaminants.

Sample container cleaning methods should be evaluated for effectiveness in removing all potential contaminants. Valves and containers should be checked for leaks after cleaning and prior to placing back in service. Sampling equipment such as separators and associated tubing should be subjected to similar cleaning procedures.

Contamination of Sample Containers and Associated Sampling Equipment

When contamination occurs during the sampling process, the sample will be non-representative. Personnel responsible for sample procurement should observe for any potential contaminants at the sample point, the sample containers, or any associated sampling equipment. If contaminants are present, the sample should be discarded, and the contaminated equipment should be cleaned.

Cleaning Procedures

The most effective cleaning agent is wet steam. Wet steam effectively removes both heavy and light contaminants. Water used in steam cleaning methods should not contain chemicals or contaminants that leave a residue in the sample container.

Evacuation methods effectively remove gasoline and lighter range contaminants but may leave residues from heavier contaminants.

A combination of steam cleaning and evacuation can remove both light and heavy contaminants.

If a floating piston container is contaminated with heavy contaminants, it may be necessary to disassemble the container for cleaning. It may also be necessary to replace the seals.

Other methods for cleaning may be used if they pass the performance criteria evaluation as described below in section “Performance Criteria for the Evaluation of Cleaning Methods.”
Leak Testing

Valve damage may occur during the cleaning process. Containers should be tested for leaks after the cleaning process. One method is to pressurize the container and immerse in water or check with a commercial leak testing solution. The leak test should ensure that there are no leaks in the container, valve connections, valve stem or valve seat.

Needle valves can leak through the valve stem packing when in either the open or closed position, but not necessarily in the opposite position. The packing should be checked for leaks with the valve in the closed position and also in the open position (while capped or plugged)

The arrows on the valves should face out from the container to prevent sampled material from leaking out of the container.

Performance Criteria for the Evaluation of Cleaning Methods

Test Solution for Cleaning Methods

Prepare a test solution for evaluation of cleaning methods by combining a mixture of 50% crude oil and 50% condensate. Mix the solution thoroughly and fill a sample container one-third full of the mixture. Leave the mixture in the container overnight. Drain the container and clean the container with the method under evaluation.

Test for Gasoline Range Contaminants

Fill the container with a gas used as the carrier gas for the gas chromatograph performing the test. The container should be heated in a manner that is consistent with the typical sample handling procedures for the facility, or at least two hours at 140 °F. The contents of the container are then analyzed by the same method used for samples by gas chromatograph to determine cleanliness. The total amount of residuals should be less than 140 parts per million by volume.

Test for Heavy Contaminants

Depressurize the sample container and remove one valve. Use a long-stemmed cotton swab to swab the length of the container. Check the swab for residues. Rotate the container 45 degrees and repeat the test seven more times. No residues should appear on the swab.

Evaluation of Cleaning Method

Any cleaning method that passes both of the above test procedures is acceptable for cleaning sample containers and related sampling equipment. The above tests can be utilized on containers in normal use that have been cleaned by the cleaning method as a form of QA/QC for the container cleaning process.

Field Cleaning Method

Field cleaning methods should be evaluated for cleaning containers and sampling equipment using the above techniques. If the field method proves effective for one type of contaminant and not both, it should be so noted. The field method would be valid only for the type contamination that it proved effective.
The data from the API MPMS Chapter 14.1 sampling project (see Annex C) clearly demonstrated that misuse of the gas sampling separator could distort the sample.

It is important for all sample system components to be clean and free from contaminants, such as water, oil, glycol, amines, free flowing hydrocarbon liquids, etc. This is equally true for separators. Refer to Annex I for cleaning procedures and for procedures to evaluate the effectiveness of cleaning procedures.

Since entrained or free-flowing hydrocarbon liquids are generally collected and accounted for in a condensate recovery system, these liquids are not desired in the vapor phase portion of the accounting system.

A gas sampling separator may help to eliminate contaminants.

These devices are intended, and should be appropriately designed, to remove contaminants from natural gas sampling systems without altering the sample quality. The objective is to remove contaminants without removing components that are part of the vapor-phase of the flowing gas stream. Contaminants include, but are not limited to, oil, glycol, amines, water and free-flowing condensed hydrocarbon liquids. Filters should be frequently checked for contamination and cleaned or replaced if necessary. Separators and filters should have a pressure rating equal to or higher than the pressure of the sample source. Heat transfer may cause condensation or vaporization when the gas temperature is depressed below or elevated above the hydrocarbon dew point.

In a system where the natural gas product is at the hydrocarbon dew point, slight changes in temperature or pressure can cause liquids to vaporize or vapors to condense.

If the change in pressure or temperature causes liquids in the sample to vaporize, the sample no longer represents the vapor phase of the flowing product stream. The sample has an artificially high concentration of heavy (high boiling point) hydrocarbons and the BTU value is elevated.

If the change in pressure or temperature causes vapor in the sample to condense, and the condensed liquid is removed prior to collection in the sample container, the sample no longer represents the vapor phase of the flowing product stream. The sample has an artificially low concentration of heavy (high boiling point) hydrocarbons and the BTU value is reduced.

In purge type sample methods, if the condensed liquids are subsequently swept back into the sample stream and collected in the sample container, the sample no longer represents the vapor phase of the flowing product stream. The sample has an artificially high concentration of heavy (high boiling point) hydrocarbons and the BTU value is elevated.

It is imperative that the gas sampling separator (See Figure J.1) be operated at the same temperature and pressure as the sample source. The drain can be left closed during the sampling process. In this case, the drain valve extension tube pigtail is not necessary. If the drain valve is opened very slightly to allow a slow weep to monitor for liquids in the sample) and the resultant cooling is so slight that the temperature of the separator is not affected, the drain valve extension tube pigtail is not necessary. If the drain valve is to be left open during the sampling process (to monitor for contaminants), the pigtail should be used.
All connections and tubing should be as short as possible, and the entire system should be free of leaks.

The separator should be insulated and have temperature indication.

If the temperature is below line temperature it will tend to remove heavy (high temperature boiling point, high heating value) components from the sample stream. If it is above line temperature, it will tend to increase the concentration of heavy components in the sample. The outlet of the separator may require supplemental heat to ensure that the product from this point to the sample container is completely in the vapor phase. See section on “Heating Requirements”. The separator is only useful for sample streams containing unwanted condensed hydrocarbon droplets, oil, amine, glycol, water or other contaminants. For clean, dry sample streams above the hydrocarbon dew point, the separator serves no useful purpose and its misuse could corrupt the sample.

![GPA Gas Separator Diagram](image)

**Figure J.1—GPA Gas Separator**

**Sampling procedure (refer to Figure J.1)**

Open the sample valve (Valve 1) at sampling point and thoroughly blow out any accumulated material. Close the sample valve (Valve 1) at the sampling point.

With the gas sampling separator inlet connected to the sample probe outlet and all valves closed, open the sample probe outlet valve.

Open the gas sampling separator inlet valve to pressure up the gas sampling separator.

If the extension tube pigtail is used, fully open the gas sampling separator drain valve. If the pigtail is not used, leave the drain valve closed or very slightly open in order to monitor for liquids in the sample as discussed above.
If the extension tube pigtail is used, slightly open the extension tube outlet valve to allow any accumulated liquids to drain. The valve should not be opened wide enough to reduce the pressure on the gas sampling separator below sample system pressure. If liquids are present, verify that the liquids are unwanted liquids and not liquids that have condensed out of the vapor portion of the sample. Ensure that the temperature and pressure are the same as flowing line temperature and pressure.

Open the gas sampling separator outlet valve and purge at a rate just fast enough to bring the gas sampling separator up to line temperature and slow enough to maintain line pressure on the gas sampling separator.

Close the gas sampling separator outlet valve.

Attach the appropriate sampling equipment to the gas sampling separator outlet valve.

Follow the selected sample procedure with the gas sampling separator outlet valve being Valve 1 for the procedure.
Annex K
(informative)

Sampling Systems for On-line Analyzers

Sampling for on-line analyzers is slightly different than spot or composite sampling. The main objective is to obtain a representative, current, clean and vapor sample at a temperature well above the vapor's hydrocarbon dew point (30°F (17°C) above the hydrocarbon dew point is recommended.) This method usually involves reducing the sample pressure at or very close to the sample probe in order to reduce sample transport lag times and reduce the likelihood of condensation of the vapor sample. Heat should be added or preserved in this method to prevent crossing of the phase boundary keeping the sample at least 30 °F (1 °C) above the hydrocarbon dew point. This reduced pressure sample will continually flow through a bypass, providing the analyzer with a current sample for analysis.

When designing on-line sample systems, consideration should be given for all sampling conditions, including plant upsets.

An example of a typical on-line analysis system is shown in the drawing below. In this example, a gas chromatograph (GC) is shown, but any on-line analyzer will work in a similar way:

![Figure L.1 Typical On-Line Chromatograph Configuration](image)

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In this graphic, the GC has an air-bath oven. However, most GC’s for natural gas measurement have mandrill ovens and do not need instrument air for heat.

In general, all sampling systems for on-line analyzers of any type have several main parts in common:

- A sample probe with membrane separator and pressure reduction (and heat, if required)
- Sample path/transport tubing
- Filtration (for protection of the analyzer from chance liquids or solids not filtered by the probe)
- A sample bypass loop (to minimize sample transport lag times)
- A sample valve loop (to provide a fresh, measured sample to the analyzer)
- At least two vents/returns per analyzed stream.

**Sampling Probes:**

It has been an industry practice that the collection end of the probe be placed within the approximate center one-third of the pipe cross-section. While it is necessary to avoid the area most likely to contain migrating liquids (the pipe wall) it may also be necessary to limit the probe length to ensure that it cannot fail due to the effects of resonant vibration. Resonant vibration can occur when the vortex shedding frequency resulting from a probe inserted into a flowing fluid is equal to or greater the probe’s natural resonant frequency. Sample probes are discussed in Section 7.

A membrane filter can be used with the probe to remove liquids from the sample.

Reduce the sample pressure to about 1 bar and compensate for any auto-refrigeration due to pressure drop that may occur by adding heat to maintain at least 30°F (17°C) above the expected hydrocarbon dew point temperature throughout the sampling process.

The sample should be installed vertically from the top of a horizontal line as is shown above. Refer to Section 7 Sample Probes.

This reduction in pressure is beneficial because a vapor that is clear of liquid at line pressure will certainly be clear of liquid when the pressure is dropped to 1 Bar.
**Sample path/transport tubing:**

The sample path/transport tubing should be as short as possible.

Sample path/transport tubing should also be oriented to keep liquid from contaminating the sample by not allowing traps or low spots.

If the temperature is expected to drop to 30°F (17°C) above the gas hydrocarbon dew point temperature, heat tracing should be used.

---

**Figure L.2 Typical Sample Probe with Regulator Installation**

**Figure L.3 Cutaway Diagram of Heat Traced Tubing.**
Filtration:

Gas samples should be free from particles as well as liquids to protect the analysis equipment from damage. Filtering should be done to the degree that the analysis equipment manufacturer specifies. This filtering should be done to prevent plugging of whatever critical orifice exists in the analyzer equipment. Many analyzers today are using "nano" technology and will require very small particle size filtration.

Filters should be located where they can be readily changed and should be checked periodically as the condition of the sample will probably demand that they be changed due to plugging up at some point. The manufacturer should be able to specify to some degree the longevity of a particular filter but, in fact, the sample cleanliness has more to do with how long they will last, and this should be of prime interest.

In-line filters will plug. The dirtier the sample the sooner they will plug. There are filters that are bypass filters and provide some self-cleaning. These provide longer life but will need to be vented in some way. The gas usage will go up as these are added to the sampling system.

Filtration methods that change the composition of the sample to be analyzed are not allowed.

Sample bypass loop:

The sampling system should use a bypass flow if the sample transport lag time requires it. A calculation as described below will indicate whether this should be done. The bypass filters mentioned in the section above will provide the restriction needed to give an excellent bypass flow. The diagram below provides a picture of how this bypass works to reduce sample transport lag times.

![Figure L.4 Typical Sample Bypass Loop](image-url)
Sample Transport Lag Time

The internal volume of the sample path/transport tubing should be calculated or at least estimated using the chart below: insert figure of tubing internal volume

<table>
<thead>
<tr>
<th>Transport Tubing Outside Diameter (inches)</th>
<th>Transport Tubing Wall Thickness (inches)</th>
<th>Volume per Linear Foot (cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/8</td>
<td>.02</td>
<td>1</td>
</tr>
<tr>
<td>¼</td>
<td>.035</td>
<td>5</td>
</tr>
<tr>
<td>3/8</td>
<td>.035</td>
<td>15</td>
</tr>
<tr>
<td>½</td>
<td>.035</td>
<td>25</td>
</tr>
</tbody>
</table>

D.8.2. The sample transport lag time should then be calculated using the following formula:

\[
t = \frac{VL}{Fs} \times \frac{P + 15}{15} \times \frac{550}{T + 450} \times \frac{1}{Zp}
\]

Where:

- \( t \) = Purge Time
- \( L \) = Line Length, ft
- \( V \) = Tubing Volume, cc/ft
- \( Fs \) = Standard Flow, cc/min
- \( P \) = Actual Pressure, PSIG
- \( T \) = Actual Temperature, °F
- \( Zp \) = Compressibility at P Pressure

Table L.1 Sample Transport Time

If the sample system has a bypass flow rotameter on it, the flow rate should be set to change the volume in the tubing three times during the expected analysis cycle time. This should provide a new sample every time an analysis is needed.

Sample valve loop and Vents/Returns

If the analysis is by gas Chromatography, the sample should be equilibrated to atmospheric pressure by means of some sort of "sample shut off" valve. This equilibration allows the very high repeatability that on-line analyzers are known for. For this reason, the bypass vent on the sample valve should not be returned to the process or to any pressure of higher or lower than the barometric. If it is necessary to capture this vent, it should probably go to a flare vent header that has minimum pressure effect on the sample equilibration.

This barometric equilibration and the fact that the entire sample is analyzed allow "normalization" of the results. This compensates for any barometric pressure changes that can occur between calibrations and gives repeatability results that can be 0.05% or better.
Bibliography


